

SUPPLEMENTARY INFORMATION

Novel anti-inflammatory agents targeting CXCR4: Design, synthesis, biological evaluation and preliminary pharmacokinetic study

Renren Bai ^a, Zhongxing Liang ^a, Younghyoun Yoon ^a, Eric Salgado ^a, Amber Feng ^a, Saumya Gurbani ^a, & Hyunsuk Shim ^{a,b,c,d,*}

^aDepartment of Radiation Oncology, School of Medicine, Emory University, Atlanta, Georgia, USA

^bWinship Cancer Institute, Emory University, Atlanta, Georgia, USA

^cDepartment of Radiology and Imaging Science, School of Medicine, Emory University, Atlanta, Georgia, USA

^dDepartment of Biomedical Engineering, Georgia Institute of Technology, Atlanta, GA 30322, USA

* Corresponding author.

Hyunsuk Shim

Department of Radiation Oncology, Emory University School of Medicine; phone, 404-778-4564; fax, 404-778-5550; E-mail, hshim@emory.edu; address, 1701 Uppergate Drive, C5018, Atlanta, GA 30322.

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Section A: Synthesis of compounds Ia-r and IIa-r.

General information

Proton and carbon NMR spectra were recorded on INOVA-400 (400 MHz), INOVA-600 (600 MHz) or VNMR-400 spectrometers at Emory NMR Research Center. The spectra obtained in CDCl_3 and $\text{DMSO}-d_6$ were referenced to the residual solvent peak. Chemical shifts (δ) are reported in parts per million (ppm) relative to residual undeuterated solvent as an internal reference. Mass spectra were recorded on a JEOL spectrometer at Emory University Mass Spectrometry Center. Analytical thin layer chromatography (TLC) was performed on precoated glass backed plates from Scientific Adsorbents Incorporated (Silica Gel 60 F254; 0.25 mm thickness).

General procedure for synthesis of intermediate 4

A solution of 4-(Boc-aminomethyl)benzylamine (**3**) (1.0 mmol) and TEA (3.0 mmol) in anhydrous DCM (8 mL) was cooled with an ice bath, then the corresponding sulfochloride (1.1 mmol, dissolved in 2 mL anhydrous DCM) was added dropwise. The reaction mixture was allowed to stir at 0 °C for 1 h. After removing the cooling bath, the resulting mixture was stirred for 5 h at room temperature, then diluted with saturated aqueous NaHCO_3 and extracted with DCM (10 mL) for three times. The combined organic layer was sequentially washed with water and brine, dried with anhydrous Na_2SO_4 , and concentrated in vacuo. The crude was purified by column chromatography with DCM/methanol (150:1, v/v) to give the product as a white solid.

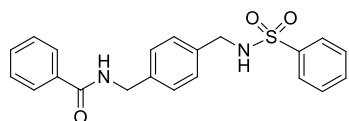
General procedure for synthesis of intermediate 5

A solution of intermediate **4** (1.0 mmol) in DCM (10 mL) was treated with trifluoroacetic acid (4 mmol) at room temperature. The resulting mixture was stirred for 8 h. The solvent was removed under reduced pressure. The residue was dissolved in saturated aqueous NaHCO_3 (2 mL) followed by adding more saturated aqueous NaHCO_3 to adjust to pH = 10. Then the mixture was filtered and the intermediate **5** was obtained as the filter cake without further purification.

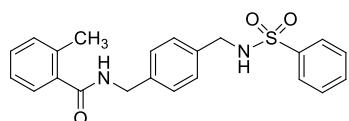
General procedure for synthesis of target compounds Ia-r and IIa-r.

A solution of intermediate **5** (1.0 mmol) and TEA (3.0 mmol) in anhydrous DCM (8 mL) was cooled with an ice bath, then the corresponding benzoyl chloride derivatives (1.1 mmol, dissolved in 2 mL anhydrous DCM) was added dropwise. The reaction mixture was allowed to stir at 0 °C for 1 h. After removing the cooling bath, the resulting mixture was stirred for 5 h at room temperature, then diluted with saturated aqueous NaHCO_3 and extracted with DCM (10 mL) three times. The combined organic layer was sequentially washed with water and brine, dried with anhydrous Na_2SO_4 , and concentrated in vacuo. The crude was purified by column chromatography with DCM/methanol to give the product as a white solid.

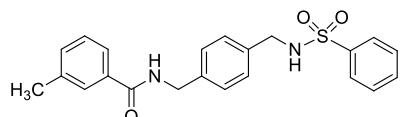
Section B: Spectroscopic data of compounds **Ia-r** and **IIa-r**.



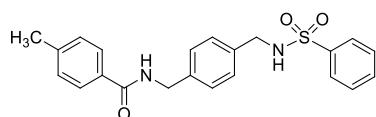
N-(4-(phenylsulfonamidomethyl)benzyl)benzamide (Ia). White solid, yield 85%, m.p. 139–141 °C. ¹H NMR (400 MHz, CDCl₃) δ 7.86–7.86 (m, 2H), 7.76–7.79 (m, 2H), 7.56–7.61 (m, 1H), 7.49–7.54 (m, 3H), 7.41–7.46 (m, 2H), 7.25–7.28 (m, 2H), 7.16–7.20 (m, 2H), 6.42 (s, 1H), 4.72 (s, 1H), 4.59 (d, *J* = 5.8 Hz, 2H), 4.13 (d, *J* = 6.2 Hz, 2H). ¹³C NMR (100 MHz, Methanol-*d*₄) δ 170.22, 142.31, 139.69, 137.69, 135.77, 133.61, 132.86, 130.25, 129.73, 129.25, 128.77, 128.44, 128.10, 47.79, 44.31. HRMS calcd for C₂₁H₂₁N₂O₃S 381.12674 [M + H]⁺, found 381.12651.



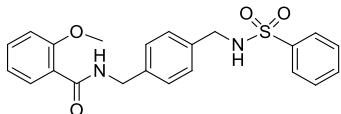
2-Methyl-N-(4-(phenylsulfonamidomethyl)benzyl)benzamide (Ib). White solid, yield 79%, m.p. 129–131 °C. ¹H NMR (400 MHz, DMSO-*d*₆) δ 8.78 (t, *J* = 6.1 Hz, 1H), 8.15 (t, *J* = 6.3 Hz, 1H), 7.79 – 7.82 (m, 2H), 7.56 – 7.66 (m, 3H), 7.31 – 7.35 (m, 2H), 7.19 – 7.26 (m, 6H), 4.39 (d, *J* = 6.1 Hz, 2H), 3.96 (d, *J* = 6.2 Hz, 2H), 2.32 (s, 3H). ¹³C NMR (100 MHz, DMSO-*d*₆) δ 168.97, 140.68, 138.60, 136.98, 136.04, 135.14, 132.29, 130.39, 129.23, 129.15, 127.53, 127.03, 126.96, 126.41, 125.47, 45.87, 42.01, 19.40. HRMS calcd for C₂₂H₂₂O₃N₂SNa 417.12433 [M + Na]⁺, found 417.12427.



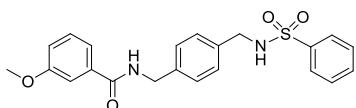
3-Methyl-N-(4-(phenylsulfonamidomethyl)benzyl)benzamide (Ic). White solid, yield 81%, m.p. 112–114 °C. ¹H NMR (400 MHz, DMSO-*d*₆) δ 8.97 (t, *J* = 6.0 Hz, 1H), 8.13 (t, *J* = 6.2 Hz, 1H), 7.78 – 7.80 (m, 2H), 7.70 (qd, *J* = 1.3, 0.7 Hz, 1H), 7.65 – 7.68 (m, 1H), 7.55 – 7.63 (m, 3H), 7.34 – 7.35 (m, 2H), 7.16 – 7.23 (m, 4H), 4.41 (d, *J* = 6.0 Hz, 2H), 3.94 (d, *J* = 6.2 Hz, 2H), 2.35 (s, 3H). ¹³C NMR (100 MHz, DMSO-*d*₆) δ 166.20, 140.66, 138.68, 137.52, 136.01, 134.33, 132.27, 131.72, 129.14, 128.16, 127.75, 127.51, 127.11, 126.41, 124.32, 45.89, 42.28, 20.93. HRMS calcd for C₂₂H₂₃O₃N₂S 395.14239 [M + H]⁺, found 395.14241.



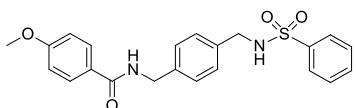
4-Methyl-N-(4-(phenylsulfonamidomethyl)benzyl)benzamide (Id). White solid, yield 87%, m.p. 188–190 °C. ¹H NMR (400 MHz, DMSO-*d*₆) δ 8.94 (t, *J* = 6.0 Hz, 1H), 8.13 (t, *J* = 6.3 Hz, 1H), 7.77 – 7.80 (m, 4H), 7.55 – 7.63 (m, 3H), 7.26 – 7.28 (m, 2H), 7.15 – 7.20 (m, 4H), 4.41 (d, *J* = 6.0 Hz, 2H), 3.94 (d, *J* = 6.2 Hz, 2H), 2.35 (s, 3H). ¹³C NMR (100 MHz, DMSO-*d*₆) δ 165.96, 141.02, 140.66, 138.74, 135.98, 132.26, 131.53, 129.13, 128.79, 127.50, 127.20, 127.10, 126.40, 45.88, 42.24, 20.92. HRMS calcd for C₂₂H₂₃N₂O₃S 395.14239 [M + H]⁺, found 395.14219.



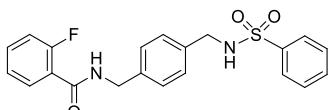
2-Methoxy-N-(4-(phenylsulfonamidomethyl)benzyl)benzamide (Ie). White solid, yield 83%, m.p. 123–125 °C. ^1H NMR (600 MHz, DMSO-*d*₆) δ 8.66 (t, *J* = 6.1 Hz, 1H), 8.12 (t, *J* = 6.3 Hz, 1H), 7.79 – 7.80 (m, 2H), 7.72 (dd, *J* = 7.7, 1.9 Hz, 1H), 7.56 – 7.63 (m, 3H), 7.47 (ddd, *J* = 9.2, 7.3, 1.9 Hz, 1H), 7.23 (d, *J* = 7.9 Hz, 2H), 7.17 (d, *J* = 7.9 Hz, 2H), 7.14 (d, *J* = 8.3 Hz, 1H), 7.03 (t, *J* = 7.4 Hz, 1H), 4.44 (d, *J* = 6.1 Hz, 2H), 3.94 (d, *J* = 6.3 Hz, 2H), 3.88 (s, 3H). ^{13}C NMR (100 MHz, DMSO-*d*₆) δ 140.66, 138.67, 135.91, 132.28, 132.10, 130.29, 129.14, 127.51, 126.94, 126.41, 123.19, 120.43, 111.96, 55.84, 45.90, 42.24. HRMS calcd for C₂₂H₂₃N₂O₄S 411.13730 [M + H]⁺, found 411.13739.



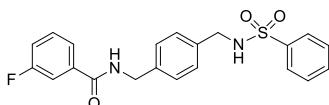
3-Methoxy-N-(4-(phenylsulfonamidomethyl)benzyl)benzamide (If). White solid, yield 78%, m.p. 120–122 °C. ^1H NMR (600 MHz, DMSO-*d*₆) δ 9.01 (t, *J* = 6.1 Hz, 1H), 8.12 (t, *J* = 6.2 Hz, 1H), 7.79 (d, *J* = 7.2 Hz, 2H), 7.56 – 7.63 (m, 3H), 7.37 – 7.47 (m, 3H), 7.22 (d, *J* = 8.0 Hz, 2H), 7.17 (d, *J* = 8.0 Hz, 2H), 7.09 (dd, *J* = 8.1, 2.7 Hz, 1H), 4.42 (d, *J* = 5.9 Hz, 2H), 3.94 (d, *J* = 6.3 Hz, 2H), 3.80 (s, 3H). ^{13}C NMR (100 MHz, DMSO-*d*₆) δ 165.80, 159.14, 140.65, 138.60, 136.04, 135.72, 132.27, 129.41, 129.14, 127.52, 127.12, 126.40, 119.41, 117.08, 112.33, 55.24, 45.88, 42.33. HRMS calcd for C₂₂H₂₃N₂O₄S 411.13730 [M + H]⁺, found 411.13721.



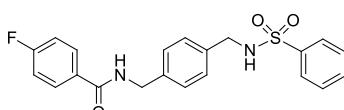
4-Methoxy-N-(4-(phenylsulfonamidomethyl)benzyl)benzamide (Ig). White solid, yield 85%, m.p. 164–166 °C. ^1H NMR (400 MHz, DMSO-*d*₆) δ 8.88 (t, *J* = 6.0 Hz, 1H), 8.13 (t, *J* = 6.3 Hz, 1H), 7.84 – 7.88 (m, 2H), 7.78 – 7.80 (m, 2H), 7.55 – 7.63 (m, 3H), 7.15 – 7.22 (m, 4H), 6.98 – 7.02 (m, 2H), 4.41 (d, *J* = 6.0 Hz, 2H), 3.94 (d, *J* = 6.3 Hz, 2H), 3.81 (s, 3H). ^{13}C NMR (100 MHz, DMSO-*d*₆) δ 165.56, 161.52, 138.85, 135.95, 132.27, 129.13, 129.00, 127.49, 127.08, 126.53, 126.40, 113.47, 55.32, 45.89, 42.22. HRMS calcd for C₂₂H₂₃N₂O₄S 411.13730 [M + H]⁺, found 411.13708.



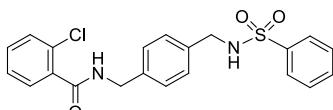
2-Fluoro-N-(4-(phenylsulfonamidomethyl)benzyl)benzamide (Ih). White solid, yield 70%, m.p. 122–124 °C. ^1H NMR (400 MHz, CDCl₃) δ 8.10–8.14 (m, 1H), 7.86–7.88 (m, 2H), 7.45–7.60 (m, 4H), 7.09–7.30 (m, 6H), 7.03 (s, 1H), 4.78 (t, *J* = 6.2 Hz, 1H), 4.62 (d, *J* = 5.6 Hz, 2H), 4.14 (d, *J* = 6.2 Hz, 2H). ^{13}C NMR (101 MHz, DMSO-*d*₆) δ 164.26, 160.66, 158.18, 140.83, 138.50, 136.46, 132.94, 132.86, 132.76, 130.36, 130.33, 129.57, 127.91, 127.42, 126.76, 124.95, 124.92, 124.21, 124.07, 116.59, 116.37, 46.18, 42.65. HRMS calcd for C₂₁H₁₉O₃N₂SFNa 421.09926 [M + Na]⁺, found 421.09950.



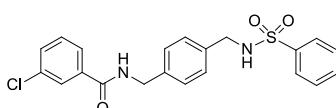
3-Fluoro-N-(4-(phenylsulfonamidomethyl)benzyl)benzamide (Ii). White solid, yield 74%, m.p. 132–134 °C. ¹H NMR (600 MHz, Chloroform-*d*) δ 7.87 (d, *J* = 7.7 Hz, 2H), 7.59 (t, *J* = 7.4 Hz, 1H), 7.49 – 7.53 (m, 4H), 7.40 (td, *J* = 8.0, 5.5 Hz, 1H), 7.17 – 7.22 (m, 5H), 6.45 (s, 1H), 4.76 (t, *J* = 6.2 Hz, 1H), 4.58 (d, *J* = 5.8 Hz, 2H), 4.13 (d, *J* = 6.3 Hz, 2H). ¹³C NMR (100 MHz, DMSO-*d*₆) δ 164.74, 163.16, 160.73, 140.66, 138.32, 136.69, 136.63, 136.14, 132.27, 130.52, 130.44, 129.14, 127.54, 127.16, 126.40, 123.37, 118.22, 118.01, 114.12, 113.89, 45.86, 42.41. HRMS calcd for C₂₁H₂₀O₃N₂SF 399.11732 [M + H]⁺, found 399.11710.



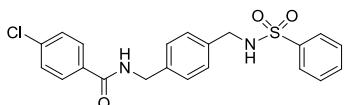
4-Fluoro-N-(4-(phenylsulfonamidomethyl)benzyl)benzamide (Ij). White solid, yield 72%, m.p. 162–164 °C. ¹H NMR (400 MHz, DMSO-*d*₆) δ 9.06 (t, *J* = 6.0 Hz, 1H), 8.13 (t, *J* = 6.3 Hz, 1H), 7.93 – 7.98 (m, 2H), 7.78 – 7.81 (m, 2H), 7.55 – 7.63 (m, 3H), 7.28 – 7.34 (m, 2H), 7.16 – 7.23 (m, 4H), 4.42 (d, *J* = 6.0 Hz, 2H), 3.94 (d, *J* = 6.3 Hz, 2H). ¹³C NMR (100 MHz, DMSO-*d*₆) δ 165.08, 165.04, 162.60, 140.66, 138.52, 136.08, 132.27, 130.79, 129.88, 129.79, 129.14, 127.53, 127.13, 126.40, 115.31, 115.09, 45.88, 42.36. HRMS calcd for C₂₁H₂₀O₃N₂SF 399.11732 [M + H]⁺, found 399.11710.



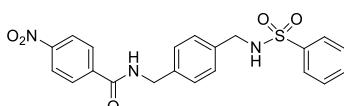
2-Chloro-N-(4-(phenylsulfonamidomethyl)benzyl)benzamide (Ik). White solid, yield 76%, m.p. 116–118 °C. ¹H NMR (600 MHz, DMSO-*d*₆) δ 8.96 (t, *J* = 5.6 Hz, 1H), 8.14 (t, *J* = 6.5 Hz, 1H), 7.81 (d, *J* = 7.7 Hz, 2H), 7.63 – 7.65 (m, 3H), 7.38 – 7.51 (m, 4H), 7.27 (d, *J* = 7.6 Hz, 2H), 7.20 (d, *J* = 7.6 Hz, 2H), 4.40 (d, *J* = 6.0 Hz, 2H), 3.96 (d, *J* = 6.2 Hz, 2H). ¹³C NMR (100 MHz, Chloroform-*d*) δ 166.58, 140.13, 137.83, 135.88, 134.95, 132.96, 131.69, 130.83, 130.53, 130.49, 129.38, 128.49, 128.40, 127.38, 127.32, 47.19, 44.00. HRMS calcd for C₂₁H₂₀O₃N₂SCl 415.08777 [M + H]⁺, found 415.08759.



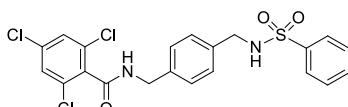
3-Chloro-N-(4-(phenylsulfonamidomethyl)benzyl)benzamide (Im). White solid, yield 80%, m.p. 114–116 °C. ¹H NMR (400 MHz, DMSO-*d*₆) δ 9.16 (t, *J* = 6.0 Hz, 1H), 8.13 (t, *J* = 6.3 Hz, 1H), 7.92 (t, *J* = 1.9 Hz, 1H), 7.84 (ddd, *J* = 7.7, 1.7, 1.1 Hz, 1H), 7.78 – 7.80 (m, 2H), 7.50 – 7.63 (m, 5H), 7.22 (d, *J* = 8.3 Hz, 2H), 7.18 (d, *J* = 8.3 Hz, 2H), 4.42 (d, *J* = 5.9 Hz, 2H), 3.94 (d, *J* = 6.3 Hz, 2H). ¹³C NMR (101 MHz, DMSO-*d*₆) δ 164.70, 140.65, 138.33, 136.28, 136.18, 133.21, 132.34, 131.13, 130.40, 129.20, 127.59, 127.22, 127.06, 126.45, 126.04, 45.90, 42.46. HRMS calcd for C₂₁H₂₀O₃N₂SCl 415.08777 [M + H]⁺, found 415.08771.



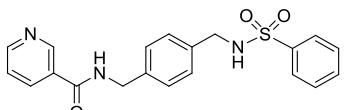
4-Chloro-N-(4-(phenylsulfonamidomethyl)benzyl)benzamide (In). White solid, yield 78%, m.p. 182–184 °C. ^1H NMR (400 MHz, DMSO-*d*₆) δ 9.12 (t, *J* = 5.9 Hz, 1H), 8.14 (t, *J* = 6.3 Hz, 1H), 7.88 – 7.92 (m, 2H), 7.78 – 7.81 (m, 2H), 7.53 – 7.63 (m, 5H), 7.15 – 7.23 (m, 4H), 4.42 (d, *J* = 4.4 Hz, 2H), 3.94 (d, *J* = 6.1 Hz, 2H). ^{13}C NMR (100 MHz, DMSO-*d*₆) δ 164.98, 140.66, 138.40, 136.12, 136.03, 133.00, 132.29, 129.15, 128.38, 127.72, 127.54, 127.15, 126.42, 45.87, 42.27. HRMS calcd for C₂₁H₁₉O₃N₂SClNa 437.06971 [M + Na]⁺, found 437.07017.



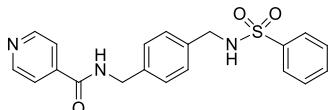
4-Nitro-N-(4-(phenylsulfonamidomethyl)benzyl)benzamide (Io). White solid, yield 70%, m.p. 201–203 °C. ^1H NMR (400 MHz, DMSO-*d*₆) δ 9.37 (t, *J* = 6.0 Hz, 1H), 8.33 (d, *J* = 8.8 Hz, 2H), 8.10 – 8.15 (m, 3H), 7.79 (d, *J* = 6.6 Hz, 2H), 7.66 – 7.52 (m, 3H), 7.24 (d, *J* = 7.9 Hz, 2H), 7.19 (d, *J* = 7.9 Hz, 2H), 4.45 (d, *J* = 5.9 Hz, 2H), 3.94 (d, *J* = 6.2 Hz, 2H). ^{13}C NMR (100 MHz, DMSO-*d*₆) δ 164.51, 149.01, 140.66, 139.94, 138.07, 136.25, 132.28, 129.14, 128.73, 127.58, 127.23, 126.40, 123.53, 45.85, 42.56. HRMS calcd for C₂₁H₂₀O₅N₃S 426.11182 [M + H]⁺, found 426.11192.



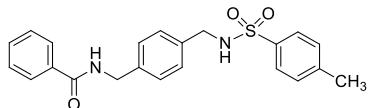
2,4,6-Trichloro-N-(4-(phenylsulfonamidomethyl)benzyl)benzamide (Ip). White solid, yield 79%, m.p. 158–160 °C. ^1H NMR (400 MHz, DMSO-*d*₆) δ 9.19 (t, *J* = 6.0 Hz, 1H), 8.15 (t, *J* = 6.3 Hz, 1H), 7.80 – 7.82 (m, 2H), 7.76 (s, 2H), 7.57 – 7.66 (m, 3H), 7.28 (d, *J* = 8.2 Hz, 2H), 7.20 (d, *J* = 8.2 Hz, 2H), 4.43 (d, *J* = 6.0 Hz, 2H), 3.96 (d, *J* = 6.3 Hz, 2H). ^{13}C NMR (100 MHz, DMSO-*d*₆) δ 162.88, 140.66, 137.42, 136.34, 135.49, 134.29, 132.31, 132.03, 129.16, 127.93, 127.48, 127.30, 126.42, 45.84, 42.13. HRMS calcd for C₂₁H₁₈O₃N₂SCl₃ 483.00982 [M + H]⁺, found 483.00996.



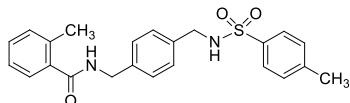
N-(4-(phenylsulfonamidomethyl)benzyl)nicotinamide (Iq). White solid, yield 60%, m.p. 109–111 °C. ^1H NMR (400 MHz, CDCl₃) δ 8.83 (dd, *J* = 2.3, 0.9 Hz, 1H), 8.69 (dd, *J* = 4.9, 1.7 Hz, 1H), 8.11 (ddd, *J* = 7.9, 2.3, 1.7 Hz, 1H), 7.86–7.90 (m, 2H), 7.57–7.62 (m, 1H), 7.50–7.54 (m, 2H), 7.36 (ddd, *J* = 8.0, 4.9, 0.9 Hz, 1H), 7.12–7.25 (m, 4H), 6.79 (t, *J* = 5.6 Hz, 1H), 5.24 (t, *J* = 6.1 Hz, 1H), 4.57 (d, *J* = 5.7 Hz, 2H), 4.11 (d, *J* = 6.1 Hz, 2H). ^{13}C NMR (101 MHz, CDCl₃) δ 165.82, 152.08, 148.20, 140.05, 137.79, 136.05, 135.49, 132.89, 130.11, 129.36, 128.58, 128.30, 127.20, 123.69, 47.09, 43.83. HRMS calcd for C₂₀H₂₀O₃N₃S 382.12199 [M + H]⁺, found 382.12154.



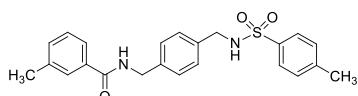
N-(4-(phenylsulfonamidomethyl)benzyl)isonicotinamide (I**r).** White solid, yield 63%, m.p. 154–156 °C. ¹H NMR (400 MHz, DMSO-*d*₆) δ 9.33 (t, *J* = 6.0 Hz, 1H), 8.73 – 8.74 (m, 2H), 8.14 (t, *J* = 6.3 Hz, 1H), 7.78 – 7.81 (m, 4H), 7.55 – 7.64 (m, 3H), 7.17 – 7.25 (m, 4H), 4.45 (d, *J* = 6.0 Hz, 2H), 3.95 (d, *J* = 6.3 Hz, 2H). ¹³C NMR (100 MHz, DMSO-*d*₆) δ 164.58, 150.25, 141.21, 140.65, 138.03, 136.25, 132.28, 129.14, 127.57, 127.19, 126.40, 121.21, 45.85, 42.41. HRMS calcd for C₂₀H₂₀O₃N₃S 382.12199 [M + H]⁺, found 382.12164.



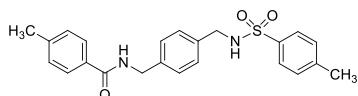
N-(4-((4-methylphenylsulfonamido)methyl)benzyl)benzamide (II**a).** White solid, yield 89%, m.p. 162–164 °C. ¹H NMR (400 MHz, CDCl₃) δ 7.75–7.79 (m, 4H), 7.49–7.53 (m, 1H), 7.41–7.46 (m, 2H), 7.27–7.33 (m, 4H), 7.18–7.20 (m, 2H), 6.41 (s, 1H), 4.62 (s, 1H), 4.60 (d, *J* = 5.7 Hz, 2H), 4.11 (d, *J* = 6.2 Hz, 2H), 2.44 (s, 3H). ¹³C NMR (100 MHz, Methanol-*d*₄) δ 170.22, 144.69, 139.66, 139.25, 137.76, 135.75, 132.84, 130.78, 129.72, 129.24, 128.74, 128.45, 128.19, 47.77, 44.32, 21.57. HRMS calcd for C₂₂H₂₂O₃N₂SNa 417.12433 [M + Na]⁺, found 417.12417.



2-Methyl-N-(4-((4-methylphenylsulfonamido)methyl)benzyl)benzamide (II**b).** White solid, yield 87%, m.p. 147–149 °C. ¹H NMR (400 MHz, DMSO-*d*₆) δ 8.79 (t, *J* = 6.1 Hz, 1H), 8.05 (t, *J* = 6.3 Hz, 1H), 7.68 – 7.71 (m, 2H), 7.38 – 7.40 (m, 2H), 7.30 – 7.36 (m, 2H), 7.19 – 7.26 (m, 6H), 4.39 (d, *J* = 6.1 Hz, 2H), 3.92 (d, *J* = 6.3 Hz, 2H), 2.38 (s, 3H), 2.32 (s, 3H). ¹³C NMR (100 MHz, DMSO-*d*₆) δ 168.98, 142.54, 138.59, 137.78, 136.99, 136.10, 135.15, 130.40, 129.57, 129.24, 127.53, 127.02, 126.97, 126.51, 125.47, 45.87, 42.03, 20.94, 19.39. HRMS calcd for C₂₃H₂₅O₃N₂S 409.15804 [M + H]⁺, found 409.15808.

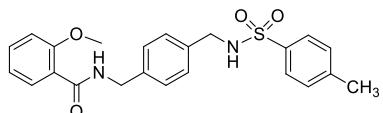


3-Methyl-N-(4-((4-methylphenylsulfonamido)methyl)benzyl)benzamide (II**c).** White solid, yield 82%, m.p. 123–125 °C. ¹H NMR (400 MHz, DMSO-*d*₆) δ 8.98 (t, *J* = 6.0 Hz, 1H), 8.03 (t, *J* = 6.3 Hz, 1H), 7.65 – 7.71 (m, 4H), 7.34 – 7.39 (m, 4H), 7.17 – 7.24 (m, 4H), 4.42 (d, *J* = 6.0 Hz, 2H), 3.90 (d, *J* = 6.3 Hz, 2H), 2.37 (s, 3H), 2.35 (s, 3H). ¹³C NMR (100 MHz, DMSO-*d*₆) δ 166.21, 142.52, 138.66, 137.76, 137.52, 136.07, 134.34, 131.72, 129.56, 128.16, 127.75, 127.51, 127.10, 126.50, 124.33, 45.88, 42.28, 20.92. HRMS calcd for C₂₃H₂₅O₃N₂S 409.15804 [M + H]⁺, found 409.15771.

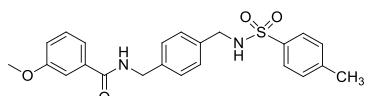


4-Methyl-N-(4-((4-methylphenylsulfonamido)methyl)benzyl)benzamide (II**d).** White solid, yield 80%, m.p. 180–182 °C. ¹H NMR (400 MHz, DMSO-*d*₆) δ 8.95 (t, *J* = 6.0 Hz, 1H), 8.03 (t, *J* = 6.3 Hz, 1H), 7.79 (d, *J* = 8.2 Hz, 2H), 7.68 (d, *J* = 8.3 Hz, 2H), 7.37 (d, *J* = 7.6 Hz, 2H), 7.27 (d, *J* = 7.6 Hz,

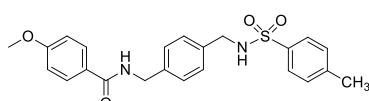
2H), 7.22 (d, J = 8.2 Hz, 2H), 7.17 (d, J = 8.3 Hz, 2H), 4.42 (d, J = 6.0 Hz, 2H), 3.90 (d, J = 6.3 Hz, 2H), 2.37 (s, 3H), 2.35 (s, 3H). ^{13}C NMR (100 MHz, DMSO- d_6) δ 165.98, 142.53, 141.03, 138.73, 136.05, 131.55, 129.56, 128.79, 127.51, 127.21, 127.09, 126.50, 45.88, 42.25, 20.92. HRMS calcd for C₂₃H₂₅N₂O₃S 409.15804 [M + H]⁺, found 409.15776.



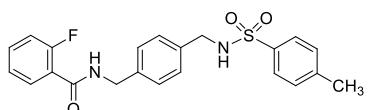
2-Methoxy-N-(4-((4-methylphenylsulfonamido)methyl)benzyl)benzamide (IIe). White solid, yield 79%, m.p. 154–156 °C. ^1H NMR (600 MHz, DMSO- d_6) δ 8.66 (t, J = 5.9 Hz, 1H), 8.03 (t, J = 6.3 Hz, 1H), 7.71–7.74 (m, 1H), 7.68 (dd, J = 8.1, 1.9 Hz, 2H), 7.47 (ddd, J = 8.2, 7.2, 1.8 Hz, 1H), 7.38 (dd, J = 8.6, 1.0 Hz, 2H), 7.24 (d, J = 8.4 Hz, 2H), 7.18 (d, J = 6.7 Hz, 2H), 7.14 (d, J = 8.4 Hz, 1H), 7.03 (td, J = 7.4, 1.1 Hz, 1H), 4.45 (d, J = 6.1 Hz, 2H), 3.91 (d, J = 6.3 Hz, 2H), 3.88 (s, 3H), 2.37 (s, 3H). ^{13}C NMR (101 MHz, DMSO- d_6) δ 165.10, 156.93, 142.60, 138.70, 137.74, 135.99, 132.16, 130.33, 129.62, 127.56, 126.97, 126.56, 123.21, 120.46, 111.97, 55.85, 45.93, 42.28, 20.98. HRMS calcd for C₂₃H₂₅N₂O₄S 425.15295 [M + H]⁺, found 425.15280.



3-Methoxy-N-(4-((4-methylphenylsulfonamido)methyl)benzyl)benzamide (IIIf). White solid, yield 82%, m.p. 130–132 °C. ^1H NMR (600 MHz, DMSO- d_6) δ 9.02 (t, J = 6.1 Hz, 1H), 8.02 (t, J = 6.3 Hz, 1H), 7.68 (d, J = 8.3 Hz, 2H), 7.36 – 7.47 (m, 5H), 7.22 (d, J = 8.2 Hz, 2H), 7.18 (d, J = 8.2 Hz, 2H), 7.09 (dd, J = 8.0, 3.0 Hz, 1H), 4.43 (d, J = 6.1 Hz, 2H), 3.90 (d, J = 6.4 Hz, 2H), 3.80 (s, 3H), 2.37 (s, 3H). ^{13}C NMR (100 MHz, DMSO- d_6) δ 165.81, 159.15, 142.53, 138.58, 137.75, 136.11, 135.73, 129.56, 129.40, 127.52, 127.11, 126.50, 119.42, 117.08, 112.34, 55.24, 45.87, 42.33, 20.92. HRMS calcd for C₂₃H₂₅N₂O₄S 425.15295 [M + H]⁺, found 425.15283.

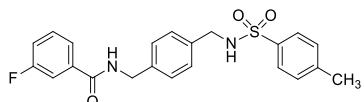


4-Methoxy-N-(4-((4-methylphenylsulfonamido)methyl)benzyl)benzamide (IIig). White solid, yield 75%, m.p. 147–149 °C. ^1H NMR (600 MHz, DMSO- d_6) δ 8.87 (t, J = 6.0 Hz, 1H), 8.02 (t, J = 6.3 Hz, 1H), 7.86 (d, J = 8.8 Hz, 2H), 7.68 (d, J = 8.3 Hz, 2H), 7.37 (d, J = 8.0 Hz, 2H), 7.21 (d, J = 8.0 Hz, 2H), 7.17 (d, J = 8.0 Hz, 2H), 6.99 (d, J = 8.8 Hz, 2H), 4.41 (d, J = 6.0 Hz, 2H), 3.90 (d, J = 6.2 Hz, 2H), 3.80 (s, 3H), 2.37 (s, 3H). ^{13}C NMR (100 MHz, DMSO- d_6) δ 165.57, 161.53, 142.52, 138.83, 137.74, 136.02, 129.55, 129.01, 127.49, 127.07, 126.53, 126.49, 113.46, 55.32, 45.88, 42.23, 20.92. HRMS calcd for C₂₃H₂₅O₄N₂S 425.15295 [M + H]⁺, found 425.15334.

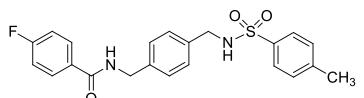


2-Fluoro-N-(4-((4-methylphenylsulfonamido)methyl)benzyl)benzamide (IIih). White solid, yield 70%, m.p. 138–140 °C. ^1H NMR (400 MHz, DMSO- d_6) δ 8.04 (t, J = 6.3 Hz, 1H), 7.67 – 7.69 (m, 2H),

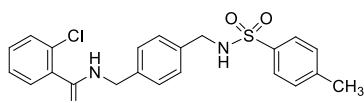
7.62 (td, $J = 7.4, 1.7$ Hz, 1H), 7.50 – 7.56 (m, 1H), 7.37 – 7.39 (m, 2H), 7.18 – 7.32 (m, 7H), 4.41 (s, 2H), 3.91 (d, $J = 6.3$ Hz, 2H), 2.38 (s, 3H). ^{13}C NMR (100 MHz, DMSO-*d*₆) δ 163.56, 160.32, 142.53, 138.18, 136.15, 132.40, 132.31, 130.03, 130.00, 129.56, 127.71, 127.54, 127.27, 127.01, 126.52, 126.50, 124.48, 124.44, 116.18, 115.96, 45.86, 42.18, 20.93. HRMS calcd for C₂₂H₂₂O₃N₂SF 413.13297 [M + H]⁺, found 413.13295.



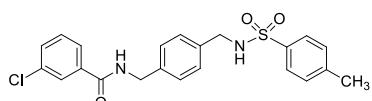
3-Fluoro-N-(4-((4-methylphenylsulfonamido)methyl)benzyl)benzamide (IIi). White solid, yield 72%, m.p. 137–139 °C. ^1H NMR (600 MHz, Chloroform-*d*) δ 7.75 (d, $J = 8.4$ Hz, 2H), 7.49 – 7.53 (m, 2H), 7.37 – 7.41 (m, 1H), 7.31 (d, $J = 7.8$ Hz, 2H), 7.25 – 7.26 (m, 2H), 7.17 – 7.21 (m, 3H), 6.51 (t, $J = 5.5$ Hz, 1H), 4.75 (t, $J = 6.2$ Hz, 1H), 4.57 (d, $J = 5.7$ Hz, 2H), 4.09 (d, $J = 6.4$ Hz, 2H), 2.44 (s, 3H). ^{13}C NMR (100 MHz, DMSO-*d*₆) δ 164.77, 163.16, 160.73, 142.53, 138.31, 137.75, 136.69, 136.63, 136.20, 130.52, 130.44, 129.56, 127.55, 127.14, 126.50, 123.38, 118.22, 118.01, 114.12, 113.90, 45.86, 42.42, 20.92. HRMS calcd for C₂₂H₂₂O₃N₂SF 413.13297 [M + H]⁺, found 413.13274.



4-Fluoro-N-(4-((4-methylphenylsulfonamido)methyl)benzyl)benzamide (IIj). White solid, yield 67%, m.p. 187–189 °C. ^1H NMR (400 MHz, DMSO-*d*₆) δ 9.06 (t, $J = 6.0$ Hz, 1H), 8.03 (t, $J = 6.3$ Hz, 1H), 7.93 – 8.00 (m, 2H), 7.66 – 7.69 (m, 2H), 7.36 – 7.37 (m, 2H), 7.28 – 7.34 (m, 2H), 7.17 – 7.24 (m, 4H), 4.43 (d, $J = 6.0$ Hz, 2H), 3.90 (d, $J = 6.3$ Hz, 2H), 2.37 (s, 3H). ^{13}C NMR (100 MHz, DMSO-*d*₆) δ 165.04, 162.61, 142.53, 138.51, 137.76, 136.14, 132.01, 130.79, 129.88, 129.79, 129.56, 127.53, 127.11, 126.50, 115.30, 115.09, 109.54, 45.87, 42.36, 20.92. HRMS calcd for C₂₂H₂₂O₃N₂SF 413.13297 [M + H]⁺, found 413.13303.

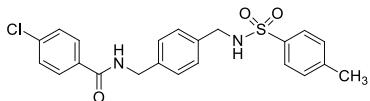


2-Chloro-N-(4-((4-methylphenylsulfonamido)methyl)benzyl)benzamide (IIk). White solid, yield 80%, m.p. 140–142 °C. ^1H NMR (600 MHz, DMSO-*d*₆) δ 8.97 (t, $J = 6.1$ Hz, 1H), 8.05 (t, $J = 6.3$ Hz, 1H), 7.70 (d, $J = 8.0$ Hz, 2H), 7.50 – 7.51 (m, 1H), 7.38 – 7.46 (m, 5H), 7.27 (d, $J = 7.8$ Hz, 2H), 7.20 (d, $J = 8.1$ Hz, 2H), 4.41 (d, $J = 6.0$ Hz, 2H), 3.92 (d, $J = 6.3$ Hz, 2H), 2.39 (s, 3H). ^{13}C NMR (100 MHz, DMSO-*d*₆) δ 166.30, 142.55, 138.06, 137.78, 136.88, 136.20, 130.72, 129.82, 129.58, 128.82, 127.52, 127.09, 127.05, 126.51, 109.53, 45.85, 42.11, 20.95. HRMS calcd for C₂₂H₂₂O₃N₂SCl 429.10342 [M + H]⁺, found 429.10349.

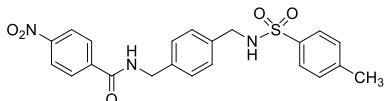


3-Chloro-N-(4-((4-methylphenylsulfonamido)methyl)benzyl)benzamide (IIm). White solid, yield 71%, m.p. 110–112 °C. ^1H NMR (400 MHz, DMSO-*d*₆) δ 9.16 (t, $J = 6.0$ Hz, 1H), 8.03 (t, $J = 6.3$ Hz,

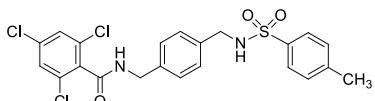
1H), 7.92 (td, $J = 1.7, 0.9$ Hz, 1H), 7.84 (ddd, $J = 7.7, 1.7, 1.1$ Hz, 1H), 7.66 – 7.69 (m, 2H), 7.61 (ddd, $J = 8.0, 2.2, 1.1$ Hz, 1H), 7.49 – 7.53 (m, 1H), 7.36 – 7.39 (m, 2H), 7.17 – 7.24 (m, 4H), 4.43 (d, $J = 5.9$ Hz, 2H), 3.90 (d, $J = 6.3$ Hz, 2H), 2.37 (s, 3H). ^{13}C NMR (100 MHz, DMSO-*d*₆) δ 164.66, 142.52, 138.26, 137.79, 136.26, 136.20, 133.16, 131.05, 130.32, 129.55, 127.54, 127.16, 127.01, 126.49, 125.98, 45.86, 42.44, 20.92. HRMS calcd for C₂₂H₂₂O₄N₃S 429.10342 [M + H]⁺, found 429.10327.



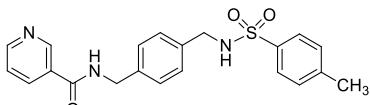
4-Chloro-N-(4-((4-methylphenylsulfonamido)methyl)benzyl)benzamide (II n). White solid, yield 71%, m.p. 201–203 °C. ^1H NMR (400 MHz, CDCl₃) δ 7.71–7.77 (m, 4H), 7.19–7.41 (m, 8H), 6.38 (s, 1H), 4.62 (s, 1H), 4.59 (d, $J = 5.8$ Hz, 2H), 4.10 (d, $J = 6.2$ Hz, 2H), 2.44 (s, 3H). ^{13}C NMR (101 MHz, DMSO-*d*₆) δ 165.75, 143.08, 138.67, 137.90, 136.52, 133.27, 131.49, 129.98, 129.48, 128.81, 127.91, 127.53, 126.84, 46.16, 42.73, 21.26. HRMS calcd for C₂₂H₂₁O₃N₂SClNa 451.08536 [M + Na]⁺, found 451.08588.



N-(4-((4-methylphenylsulfonamido)methyl)benzyl)-4-nitrobenzamide (II o). White solid, yield 66%, m.p. 207–209 °C. ^1H NMR (400 MHz, DMSO-*d*₆) δ 9.37 (t, $J = 6.0$ Hz, 1H), 8.31 – 8.34 (m, 2H), 8.02 – 8.12 (m, 2H), 8.04 (t, $J = 6.3$ Hz, 1H), 7.68 (d, $J = 8.2$ Hz, 2H), 7.38 (d, $J = 8.0$ Hz, 2H), 7.25 (d, $J = 8.0$ Hz, 2H), 7.19 (d, $J = 8.0$ Hz, 2H), 4.46 (d, $J = 5.9$ Hz, 2H), 3.91 (d, $J = 6.3$ Hz, 2H), 2.37 (s, 3H). ^{13}C NMR (100 MHz, DMSO-*d*₆) δ 164.51, 149.00, 142.53, 139.94, 138.05, 137.73, 136.31, 129.56, 128.73, 127.58, 127.21, 126.49, 123.53, 45.84, 42.57, 20.93. HRMS calcd for C₂₂H₂₂N₃O₅S 440.12747 [M + H]⁺, found 440.12768.

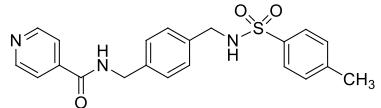


2,4,6-Trichloro-N-(4-((4-methylphenylsulfonamido)methyl)benzyl)benzamide (II p). White solid, yield 77%, m.p. 188–190 °C. ^1H NMR (400 MHz, DMSO-*d*₆) δ 9.19 (t, $J = 6.0$ Hz, 1H), 8.05 (t, $J = 6.3$ Hz, 1H), 7.76 (s, 2H), 7.68 – 7.70 (m, 2H), 7.38 – 7.40 (m, 2H), 7.29 (d, $J = 8.2$ Hz, 2H), 7.20 (d, $J = 8.2$ Hz, 2H), 4.43 (d, $J = 6.0$ Hz, 2H), 3.92 (d, $J = 6.3$ Hz, 2H), 2.39 (s, 3H). ^{13}C NMR (100 MHz, DMSO-*d*₆) δ 162.88, 142.56, 137.76, 137.40, 136.39, 135.49, 134.29, 132.03, 129.58, 127.93, 127.48, 127.28, 126.51, 45.84, 42.12, 20.95. HRMS calcd for C₂₂H₂₀N₂O₃SCl₃ 497.02547 [M + H]⁺, found 497.02593.



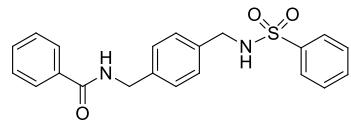
N-(4-((4-methylphenylsulfonamido)methyl)benzyl)nicotinamide (II q). White solid, yield 65%, m.p. 120–122 °C. ^1H NMR (400 MHz, Chloroform-*d*) δ 8.86 (d, $J = 1.8$ Hz, 1H), 8.70 (dd, $J = 4.9, 1.8$ Hz, 1H), 8.11 (dt, $J = 7.9, 2.0$ Hz, 1H), 7.75–7.78 (m, 2H), 7.35–7.38 (m, 1H), 7.27–7.33 (m, 3H),

7.18–7.25 (m, 3H), 6.75 (t, J = 5.9 Hz, 1H), 5.07 (t, J = 6.1 Hz, 1H), 4.59 (d, J = 5.7 Hz, 2H), 4.09 (d, J = 6.1 Hz, 2H), 2.44 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 165.86, 152.19, 148.30, 143.70, 137.82, 137.10, 136.16, 135.35, 130.08, 129.96, 128.56, 128.30, 128.27, 123.62, 47.07, 43.84, 21.72. HRMS calcd for $\text{C}_{21}\text{H}_{22}\text{O}_3\text{N}_6\text{S}$ 396.13764 [$\text{M} + \text{H}]^+$, found 396.13734.

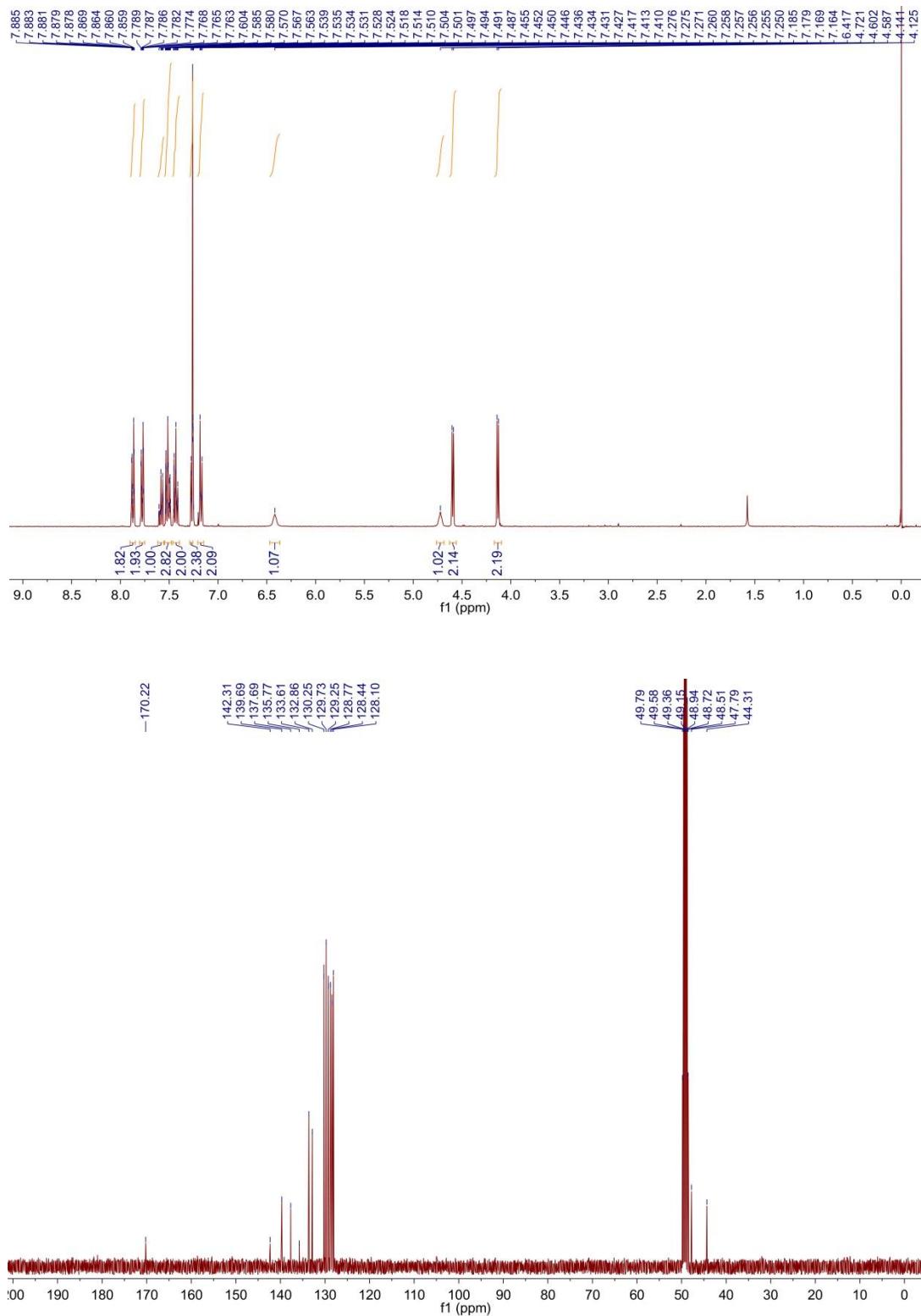


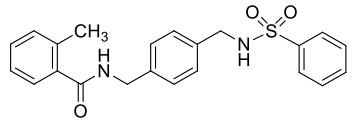
N-(4-((4-methylphenylsulfonamido)methyl)benzyl)isonicotinamide (IIr). White solid, yield 63%, m.p. 163–165 °C. ^1H NMR (400 MHz, $\text{DMSO}-d_6$) δ 9.33 (t, J = 6.0 Hz, 1H), 8.72 – 8.74 (m, 2H), 8.04 (t, J = 6.3 Hz, 1H), 7.78 – 7.79 (m, 2H), 7.67 – 7.70 (m, 2H), 7.37 – 7.39 (m, 2H), 7.16 – 7.25 (m, 4H), 4.45 (d, J = 6.0 Hz, 2H), 3.91 (d, J = 6.3 Hz, 2H), 2.37 (s, 3H). ^{13}C NMR (100 MHz, $\text{DMSO}-d_6$) δ 164.58, 150.24, 142.53, 141.21, 138.01, 137.75, 136.31, 129.56, 127.58, 127.18, 126.50, 121.22, 45.84, 42.42, 20.92. HRMS calcd for $\text{C}_{21}\text{H}_{22}\text{O}_3\text{N}_3\text{S}$ 396.13764 [$\text{M} + \text{H}]^+$, found 396.13739.

Section C: ^1H and ^{13}C NMR Spectras of compounds Ia-r and IIa-r.

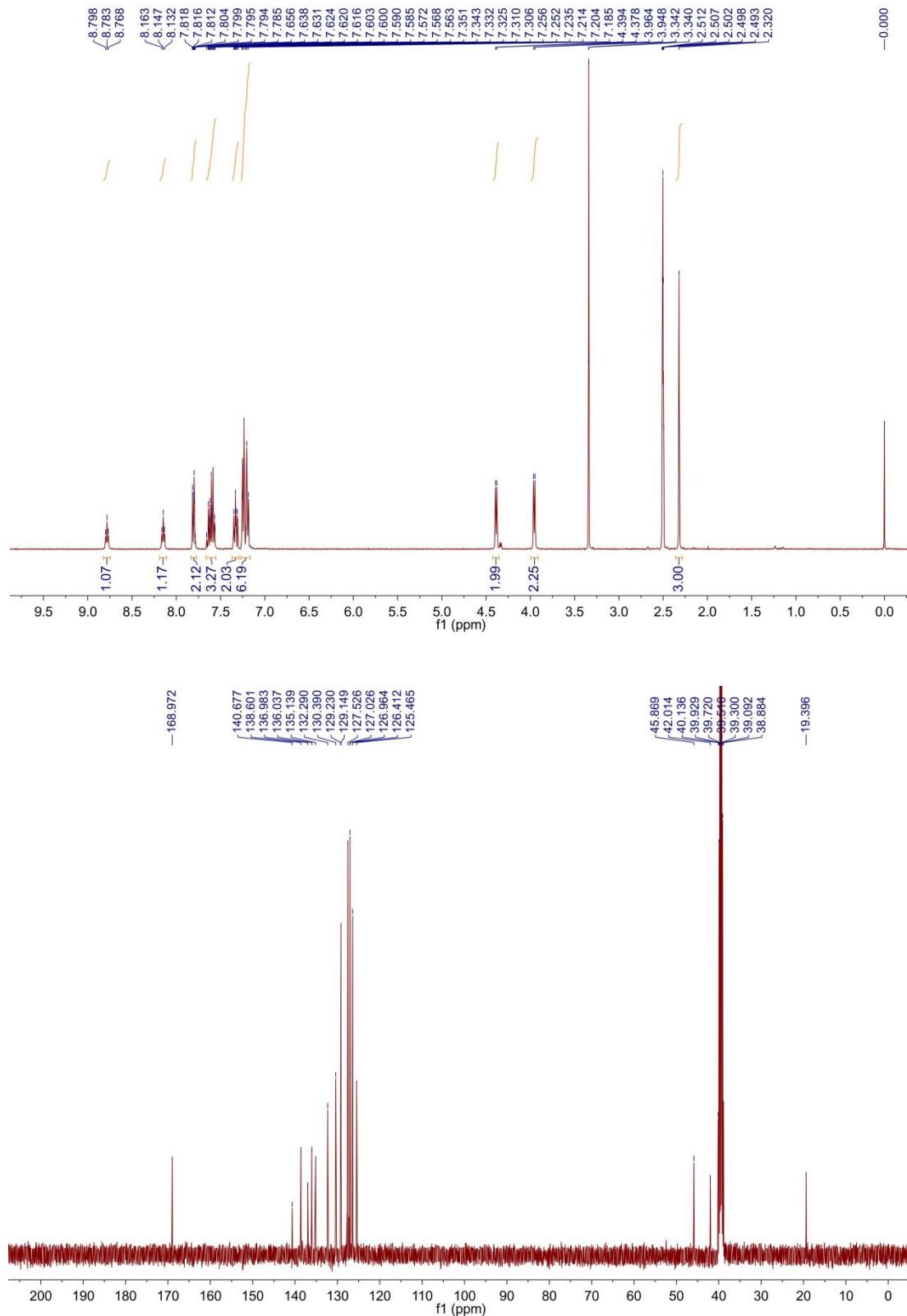


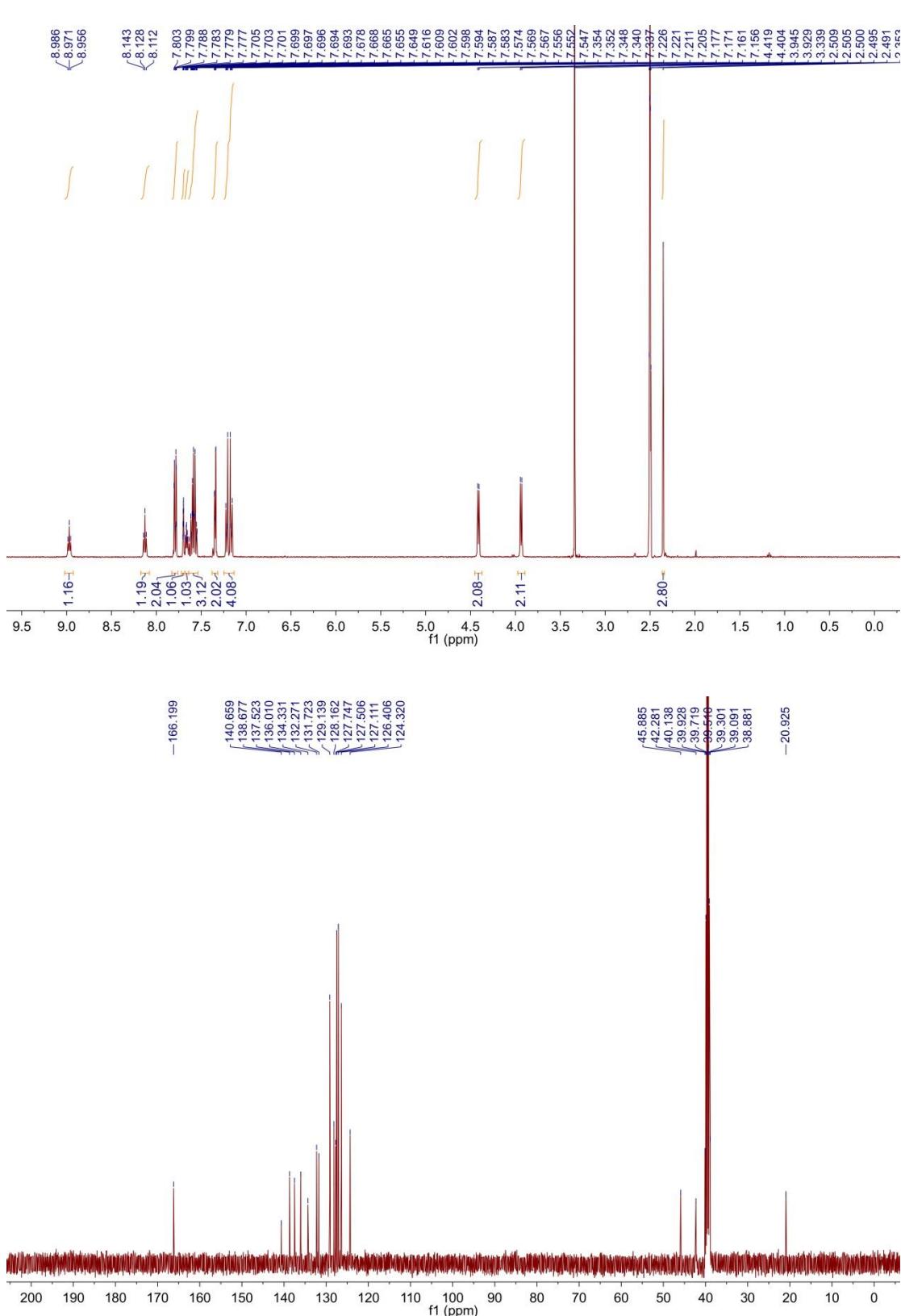
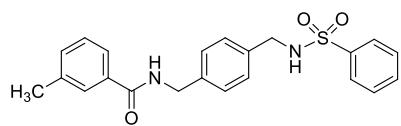
N-(4-(phenylsulfonamidomethyl)benzyl)benzamide (Ia).

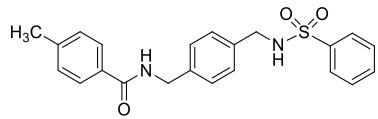




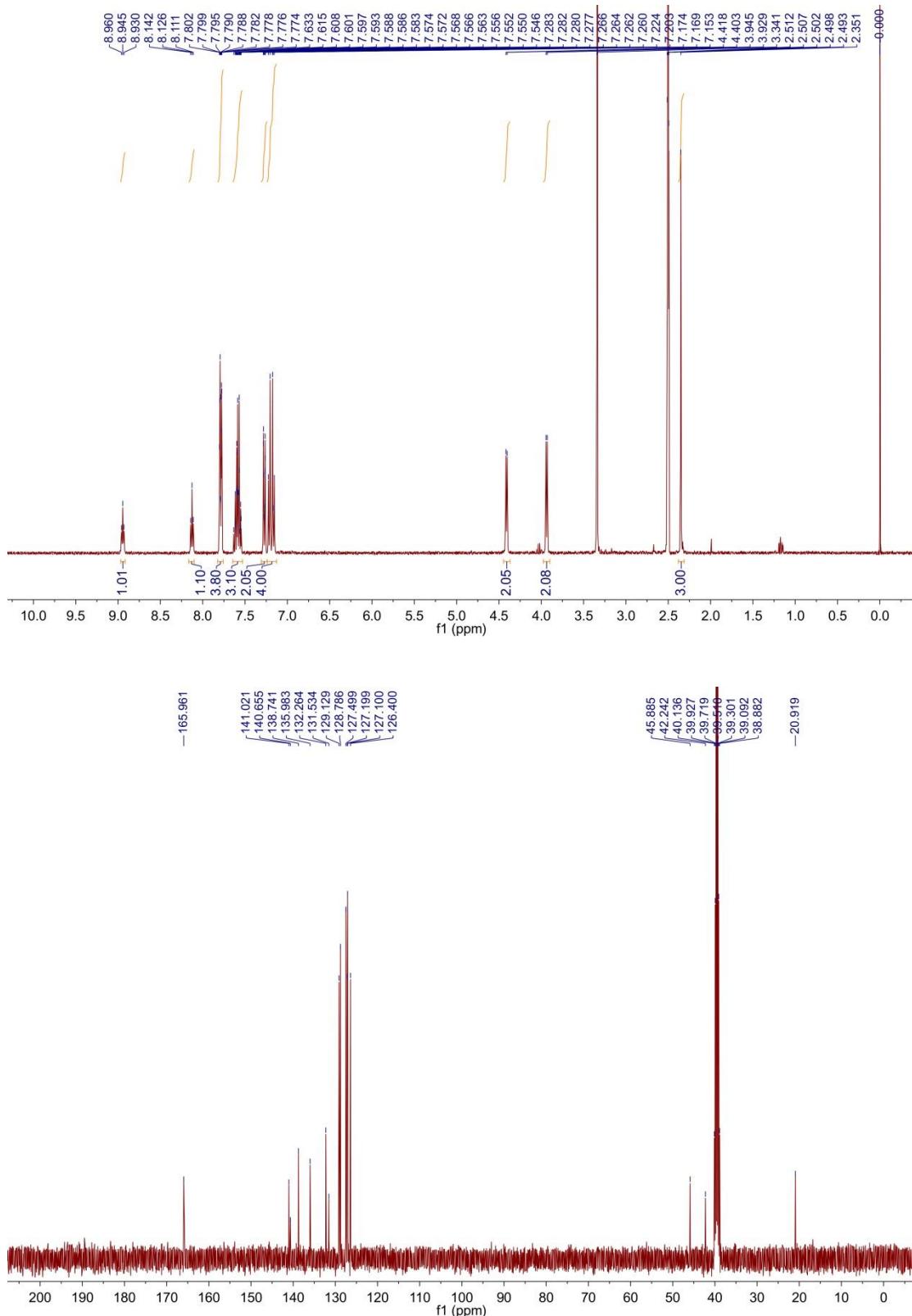
2-Methyl-N-(4-(phenylsulfonamidomethyl)benzyl)benzamide (Ib).

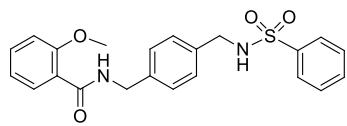




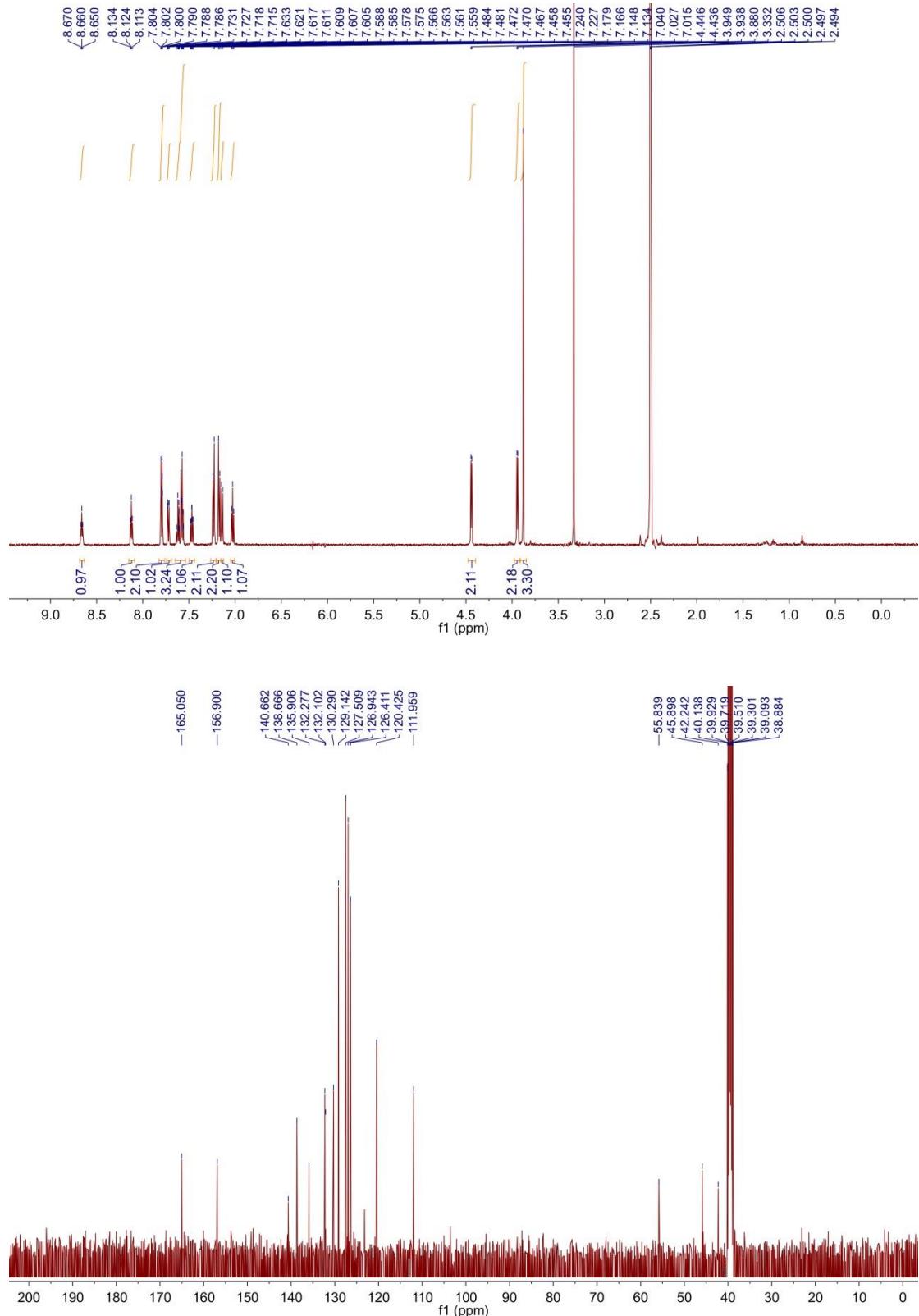


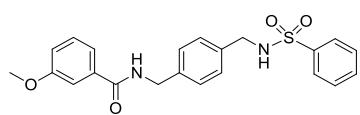
4-Methyl-N-(4-(phenylsulfonamidomethyl)benzyl)benzamide (Id).



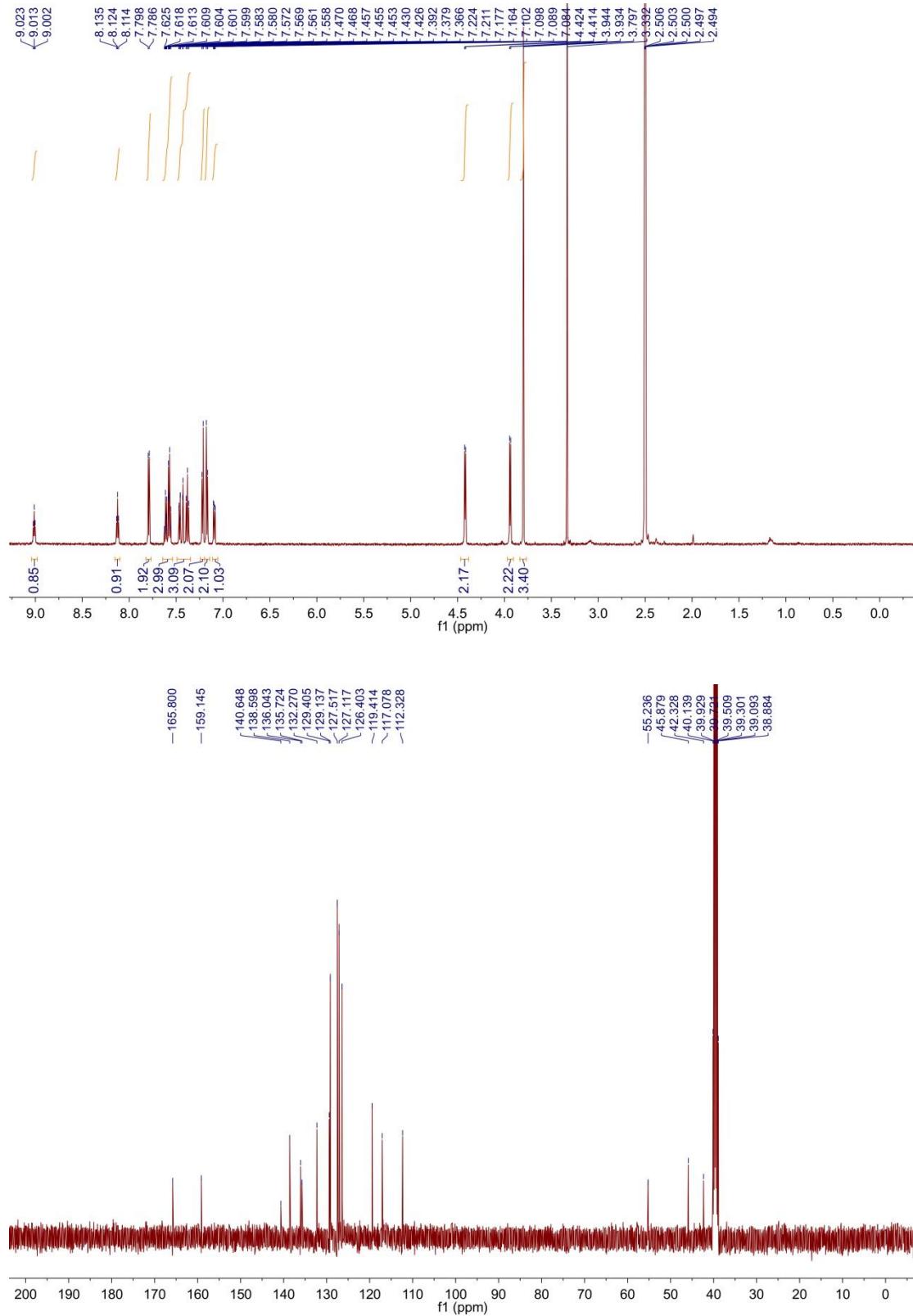


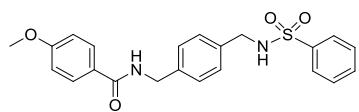
2-Methoxy-N-(4-(phenylsulfonamidomethyl)benzyl)benzamide (Ie).



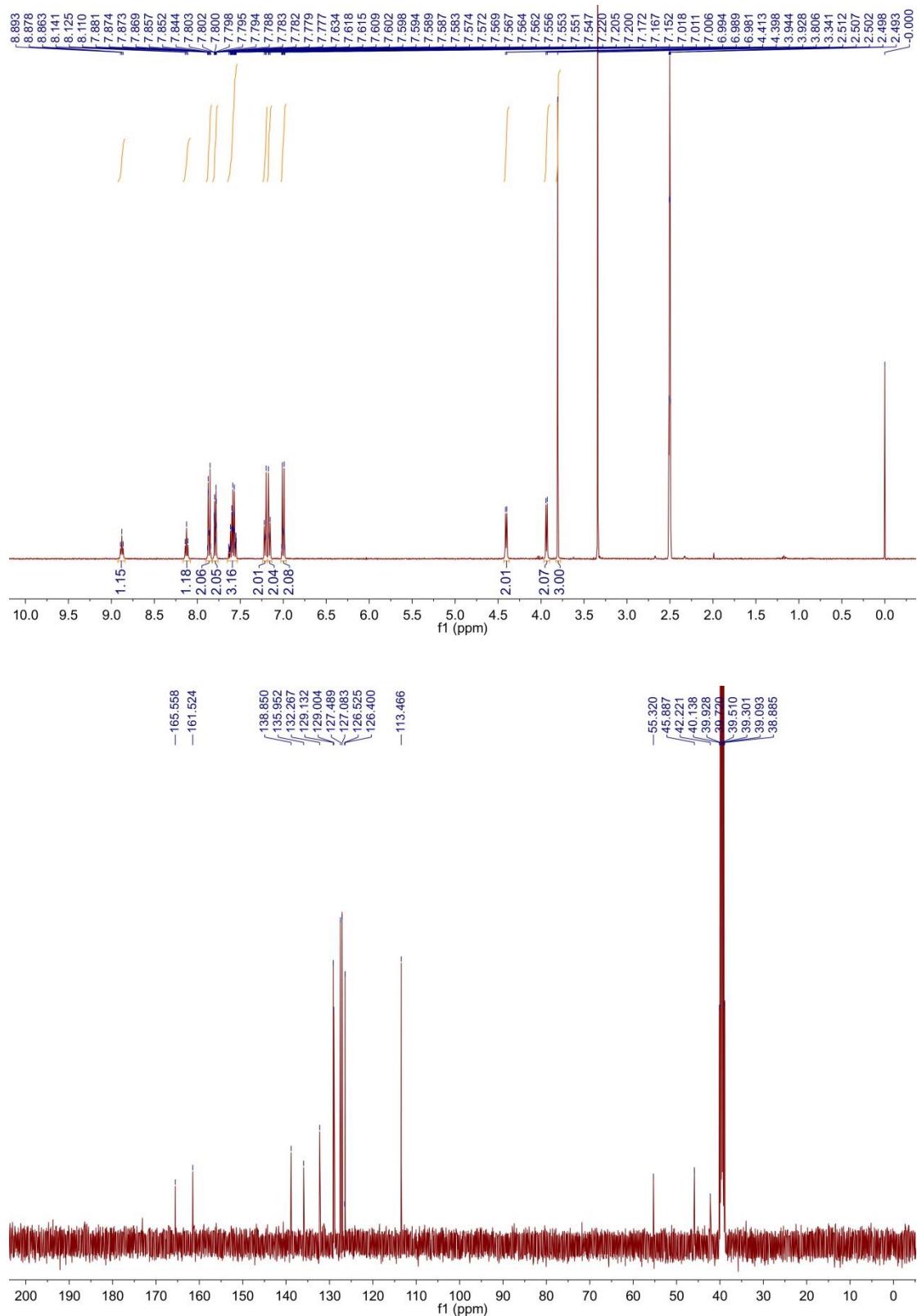


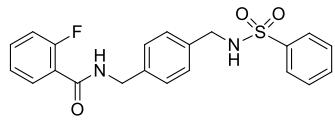
3-Methoxy-N-(4-(phenylsulfonamidomethyl)benzyl)benzamide (If).



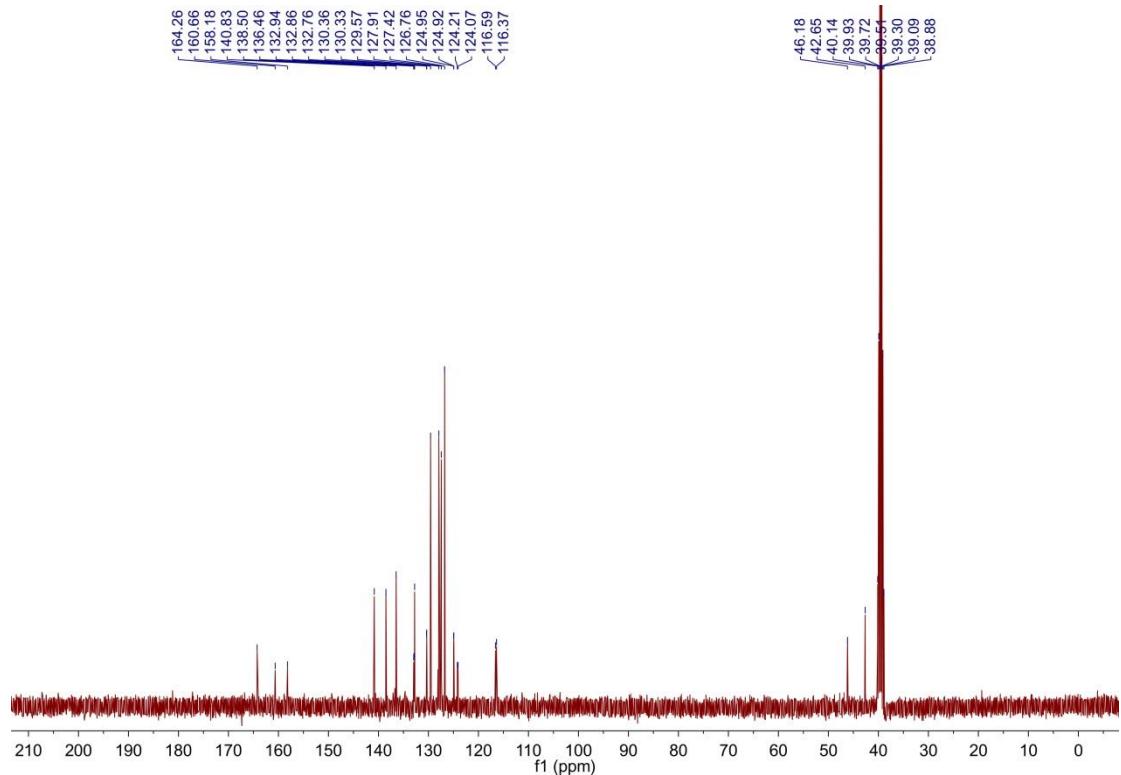
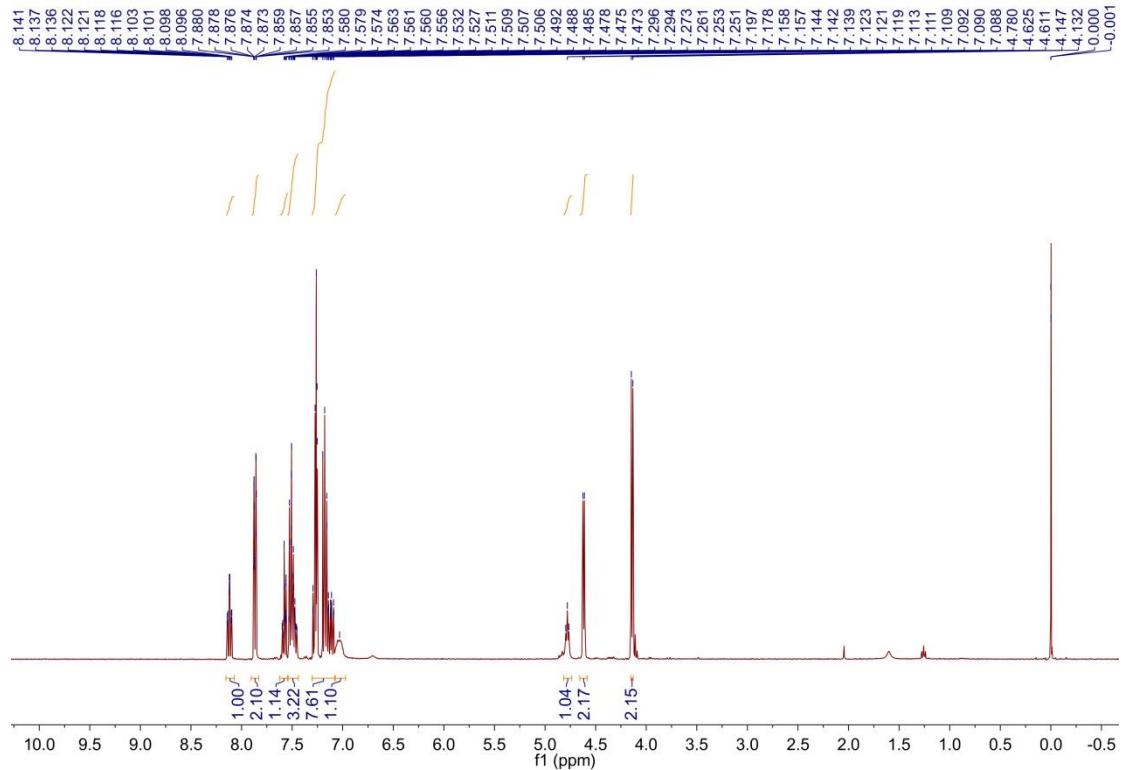


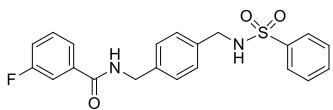
4-Methoxy-N-(4-(phenylsulfonamidomethyl)benzyl)benzamide (Ig).



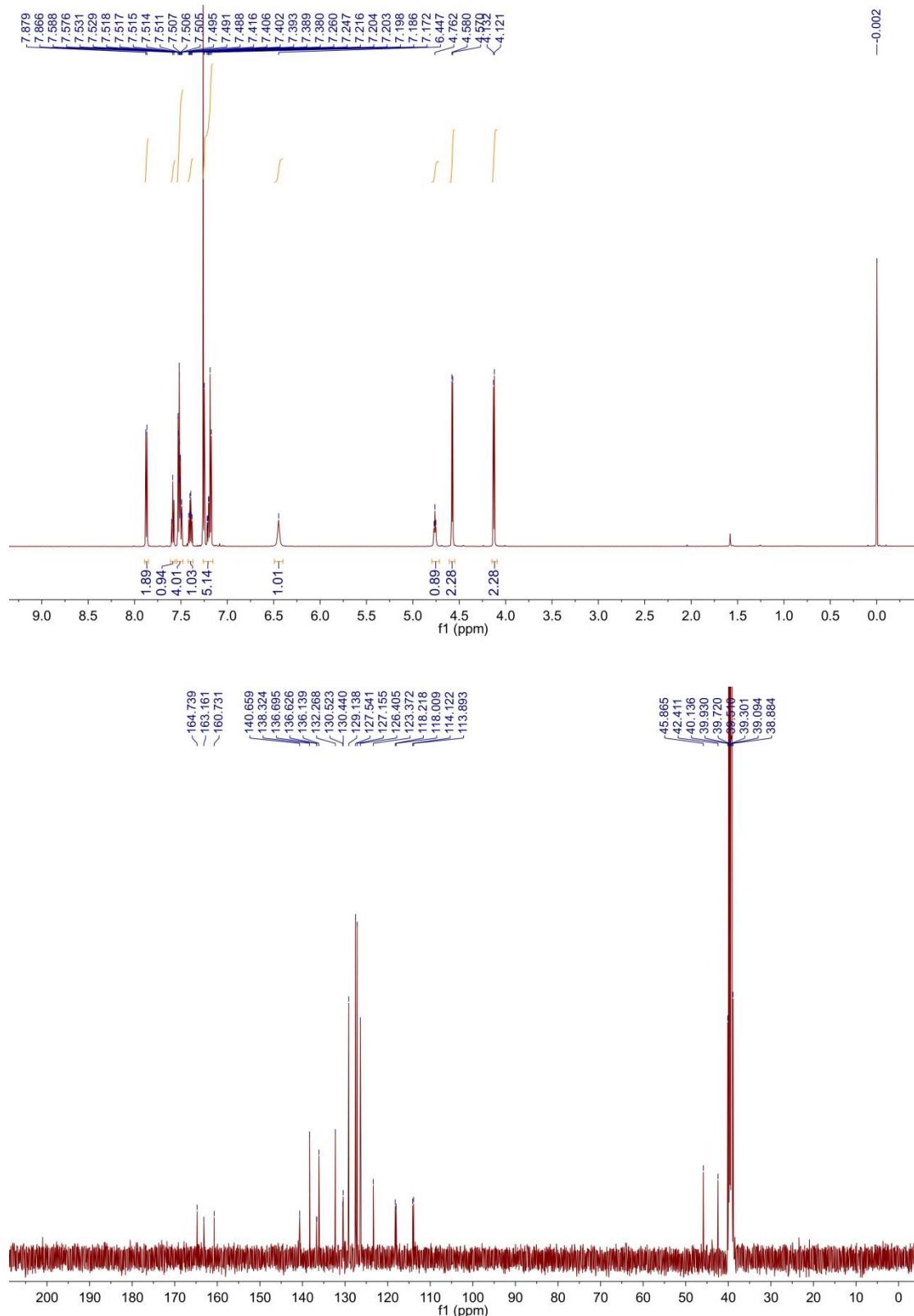


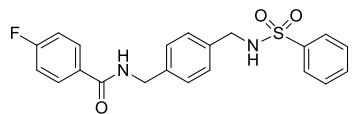
2-Fluoro-N-(4-(phenylsulfonamidomethyl)benzyl)benzamide (Ih).



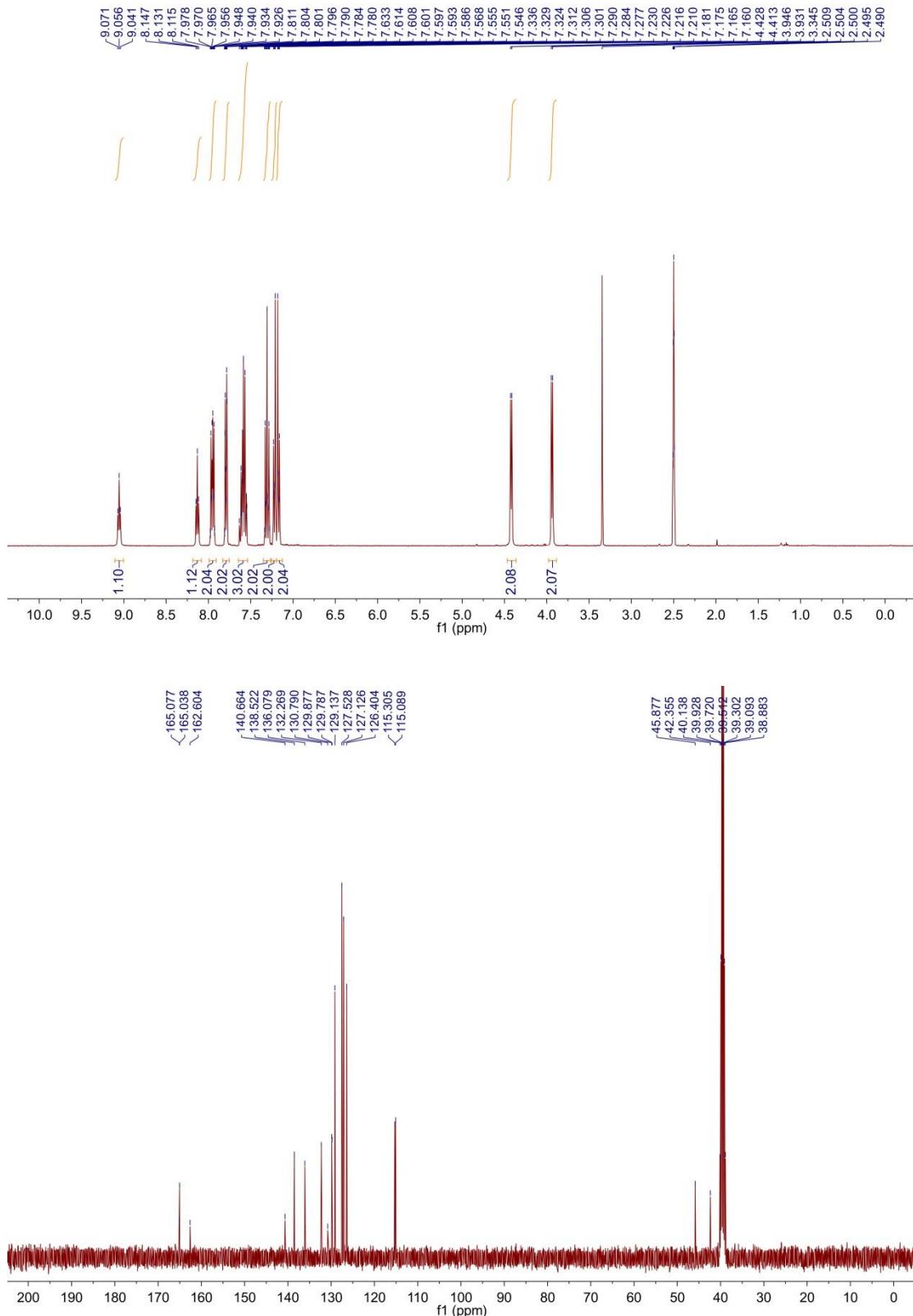


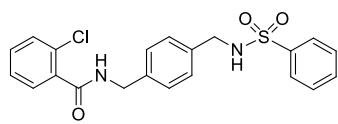
3-Fluoro-N-(4-(phenylsulfonamidomethyl)benzyl)benzamide (II).



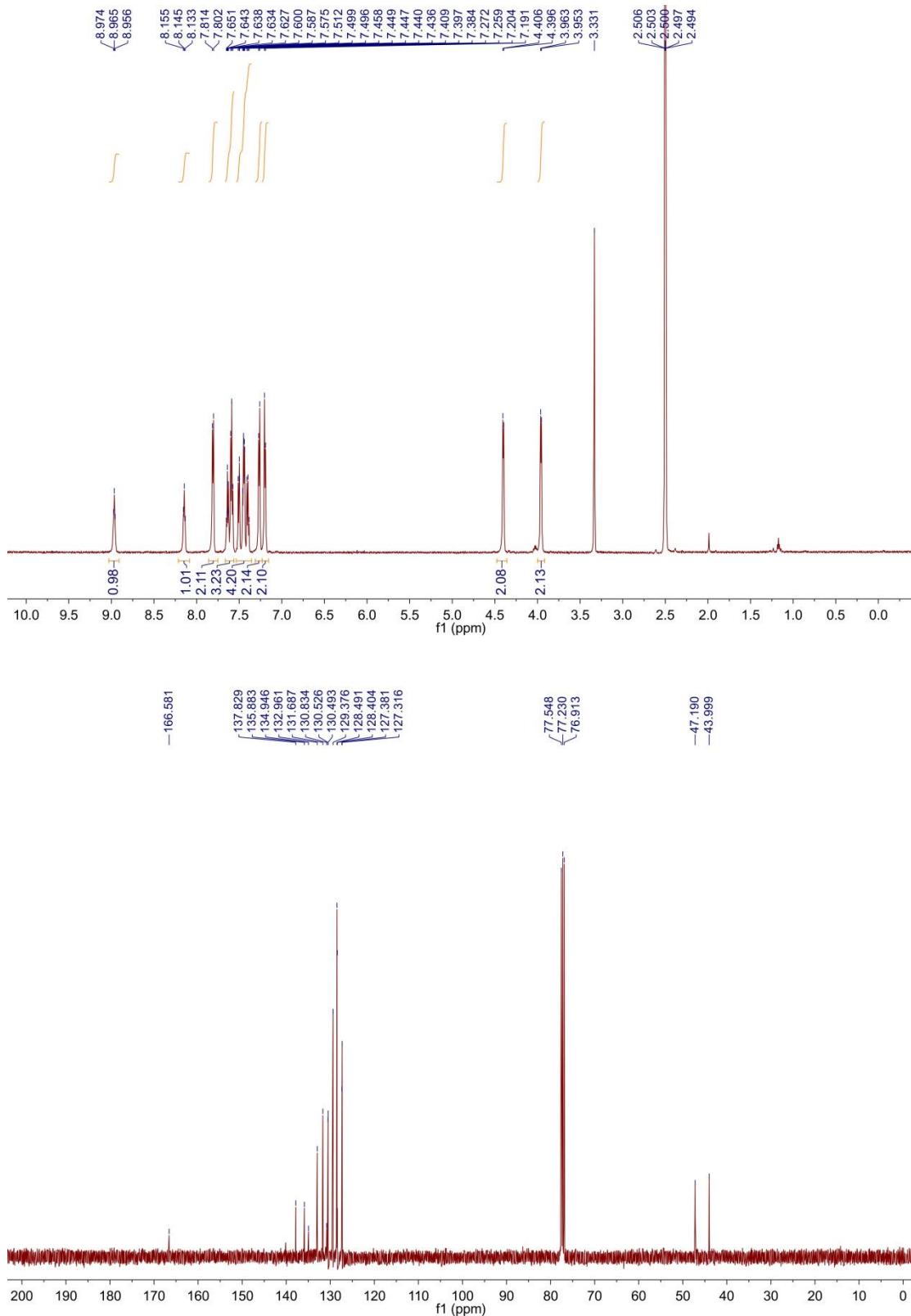


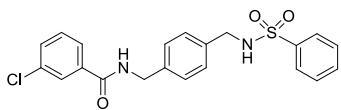
4-Fluoro-N-(4-(phenylsulfonamidomethyl)benzyl)benzamide (Ij).



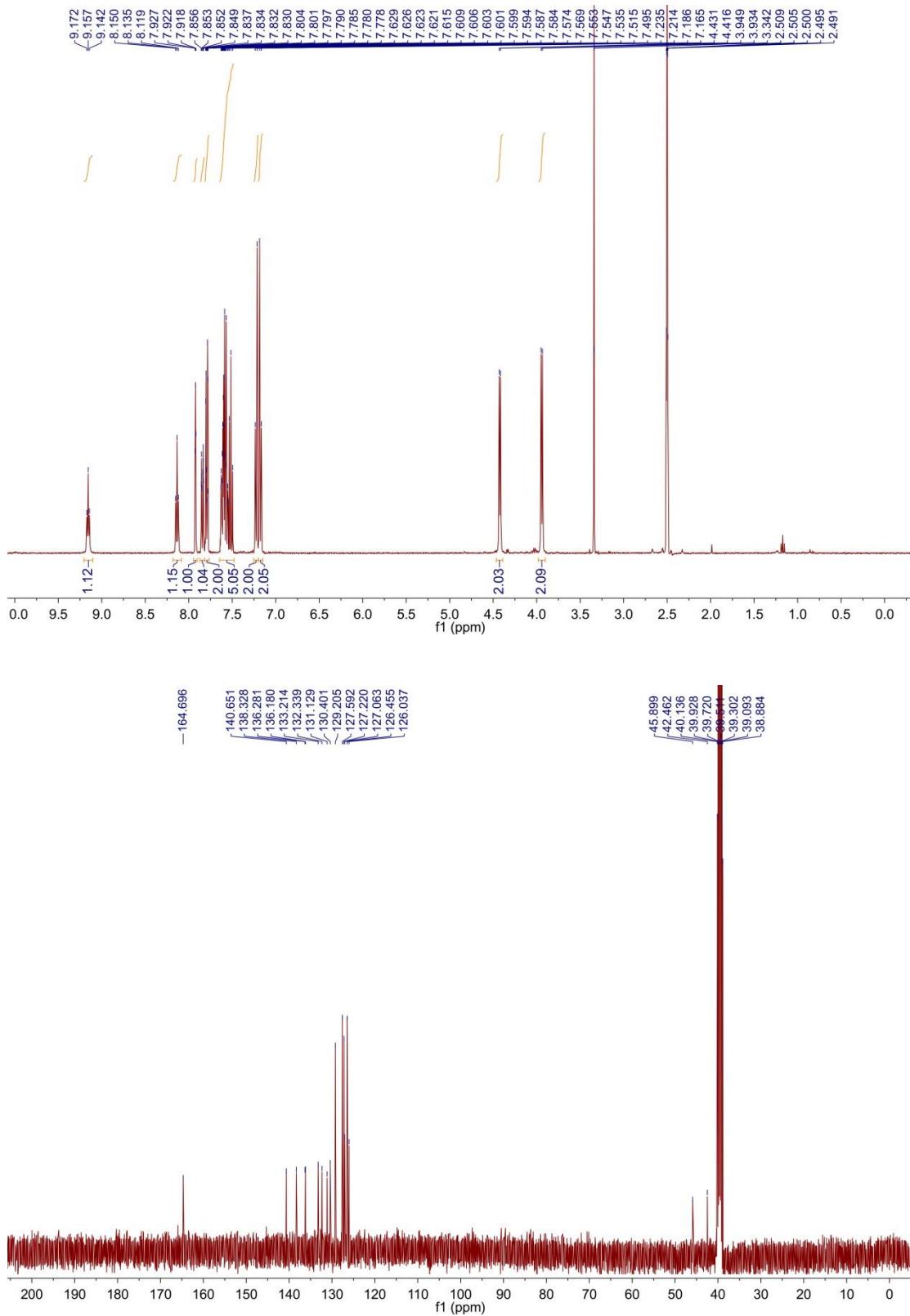


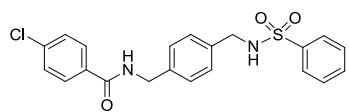
2-Chloro-N-(4-(phenylsulfonamidomethyl)benzyl)benzamide (Ik).



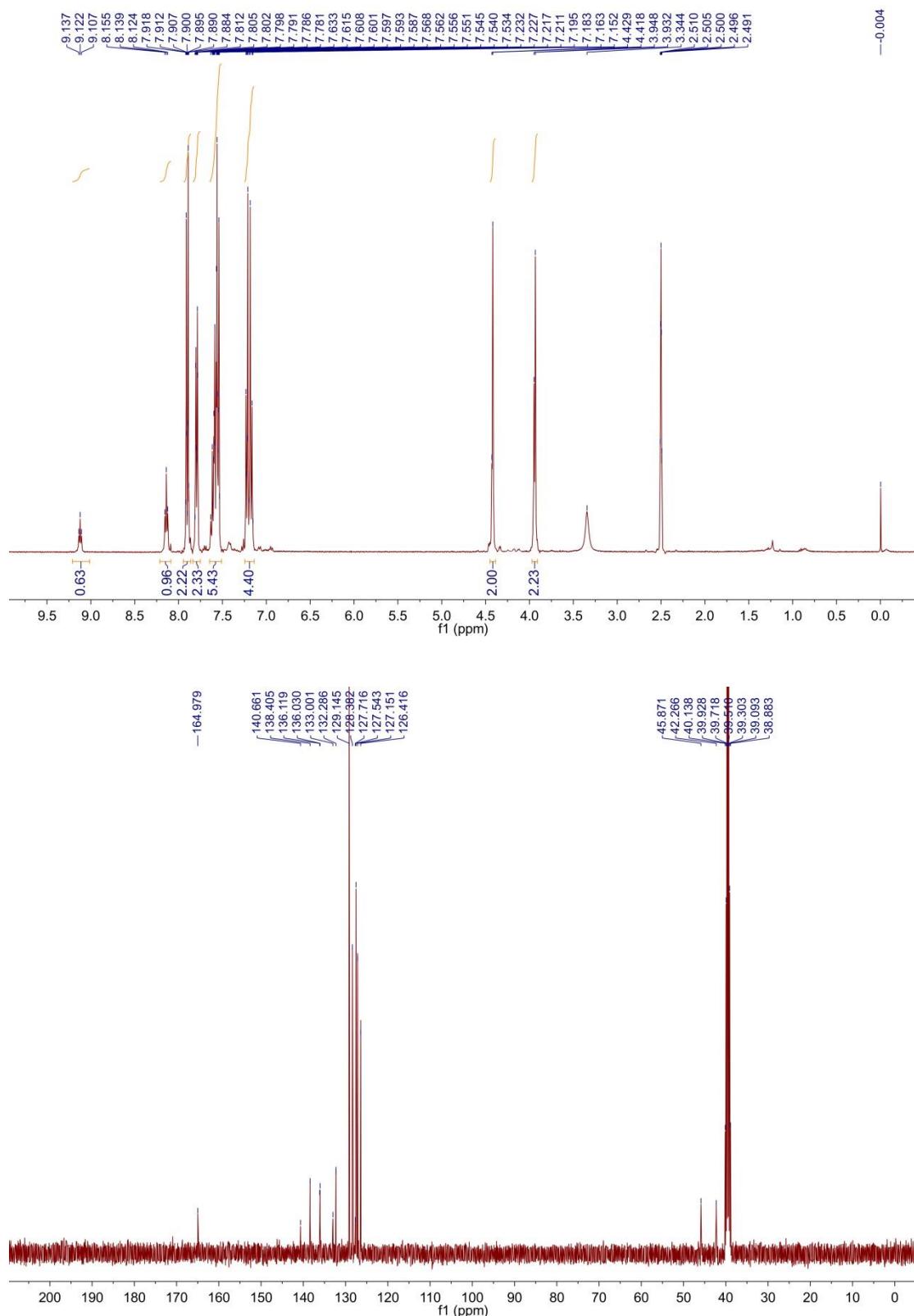


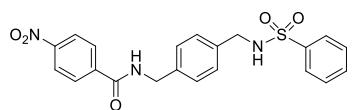
3-Chloro-N-(4-(phenylsulfonamidomethyl)benzyl)benzamide (Im).



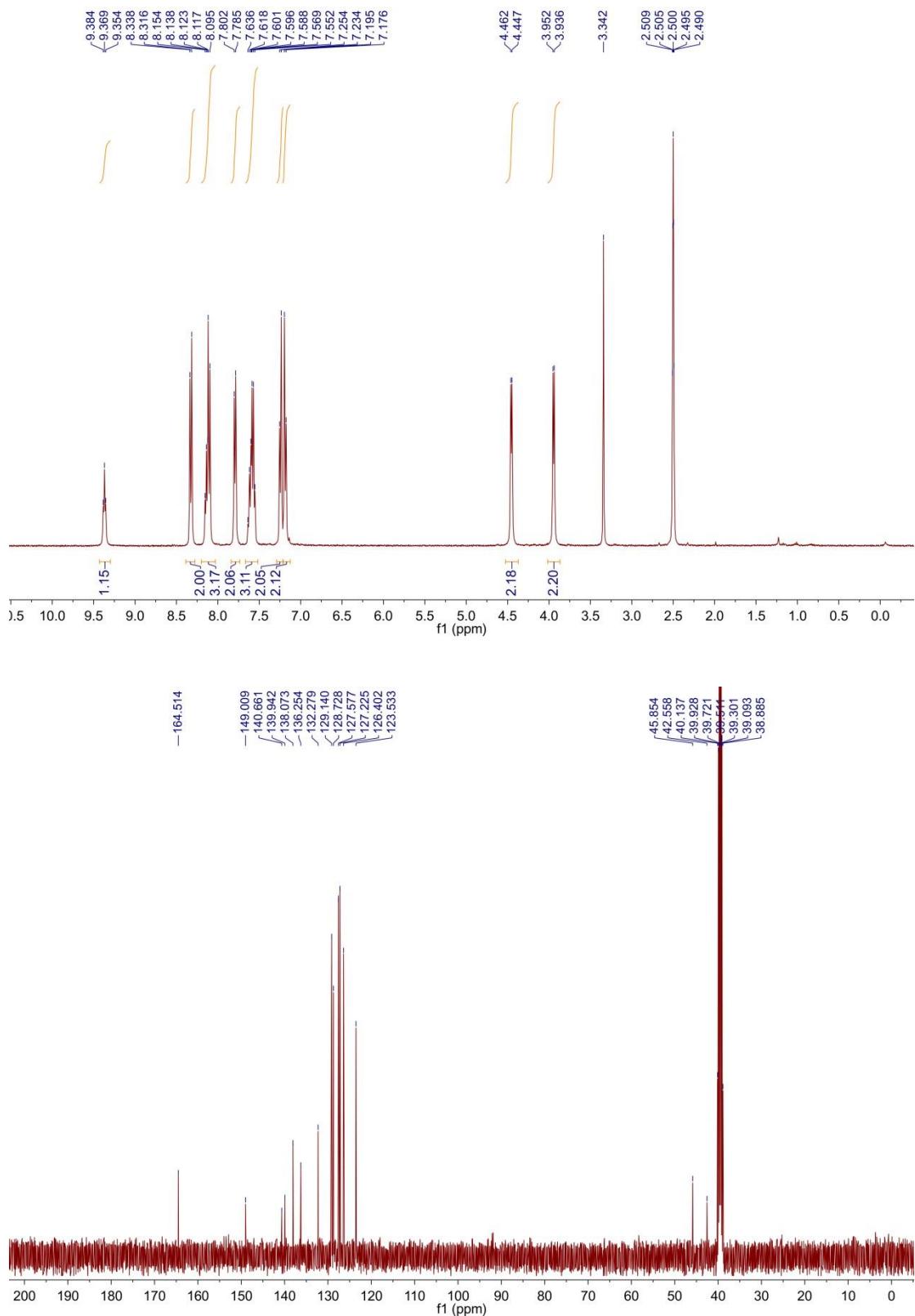


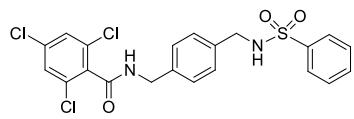
4-Chloro-N-(4-(phenylsulfonamidomethyl)benzyl)benzamide (In).



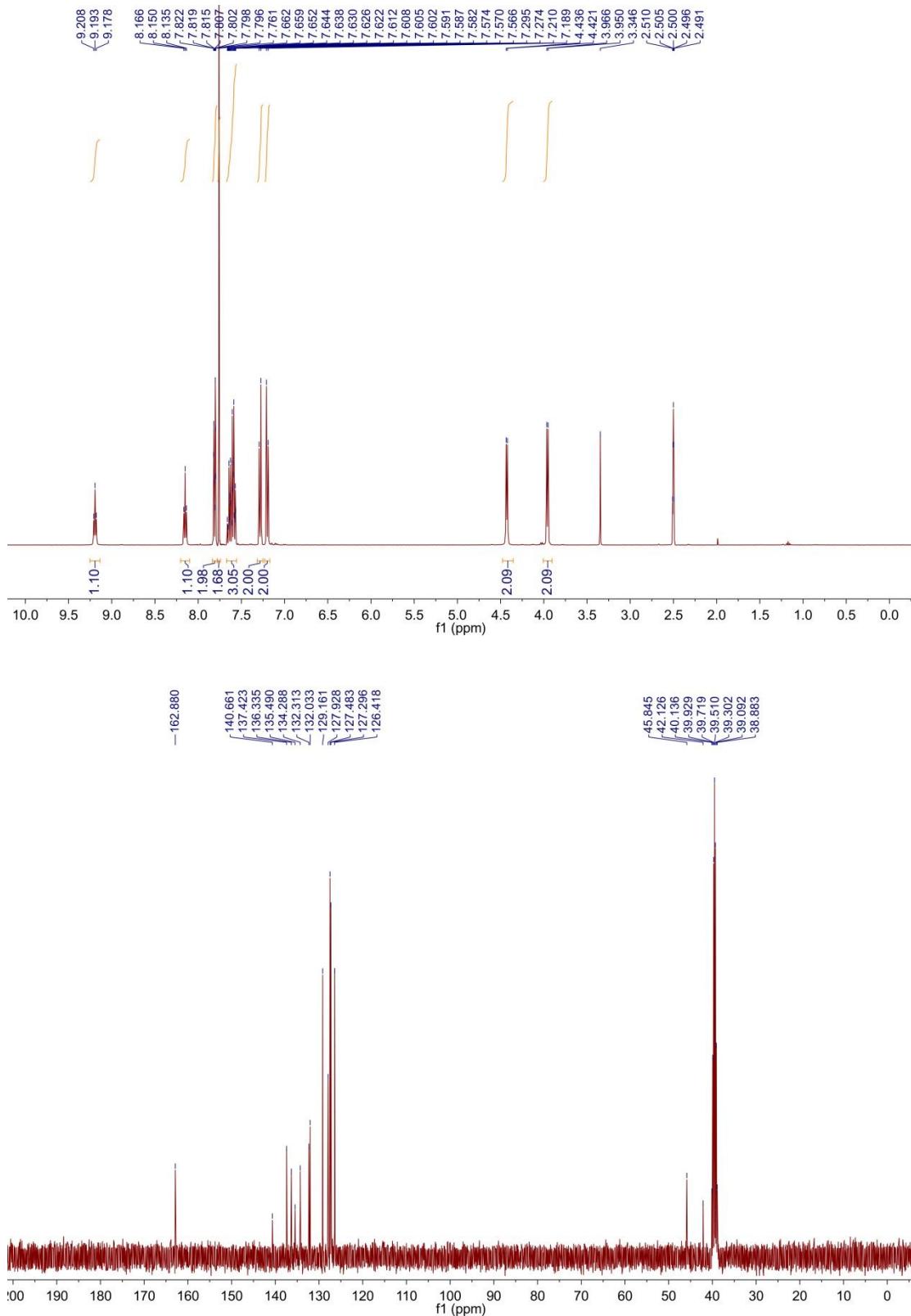


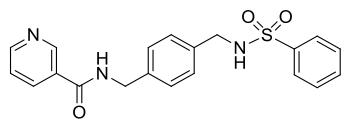
4-Nitro-N-(4-(phenylsulfonamidomethyl)benzyl)benzamide (I_o).



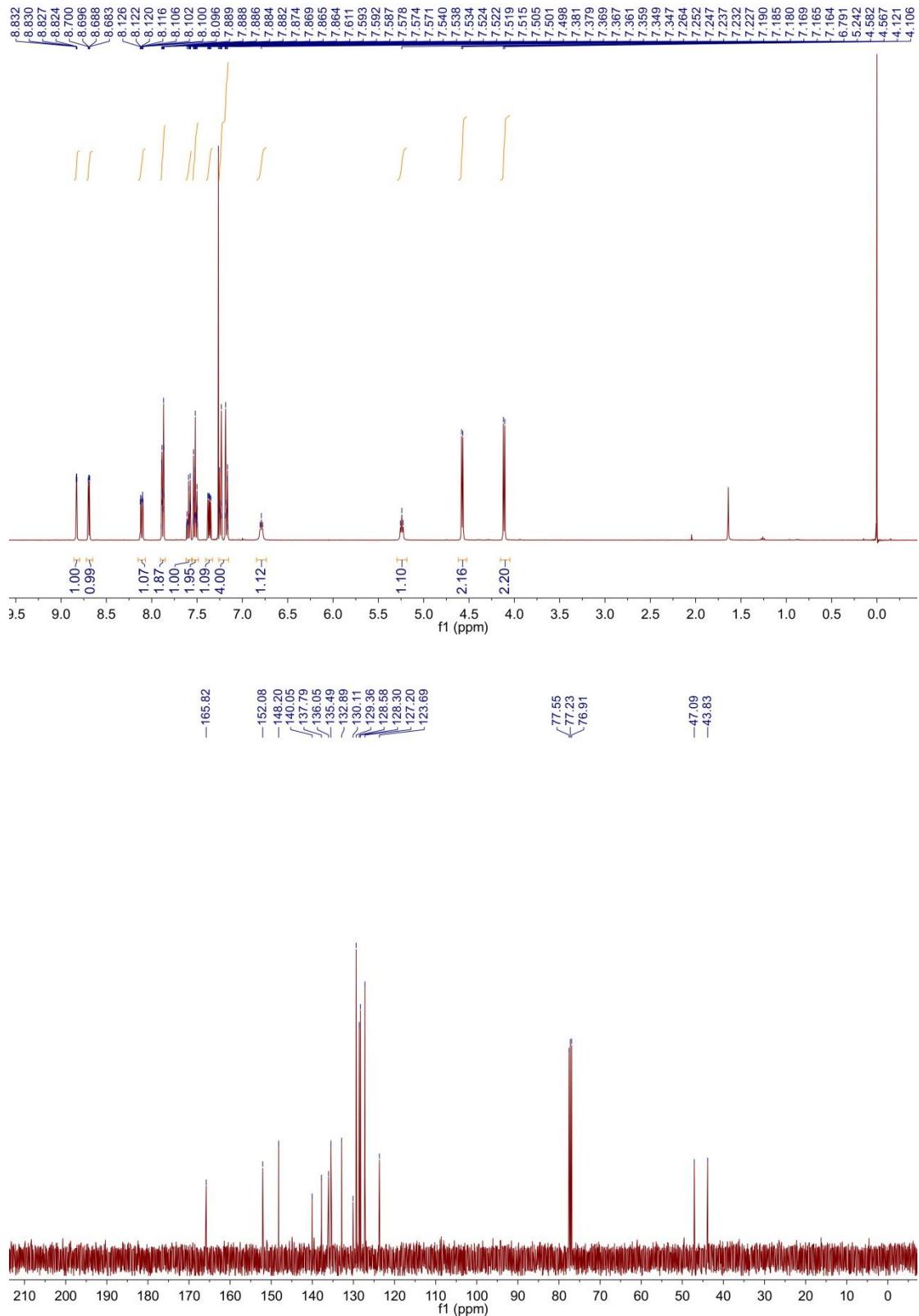


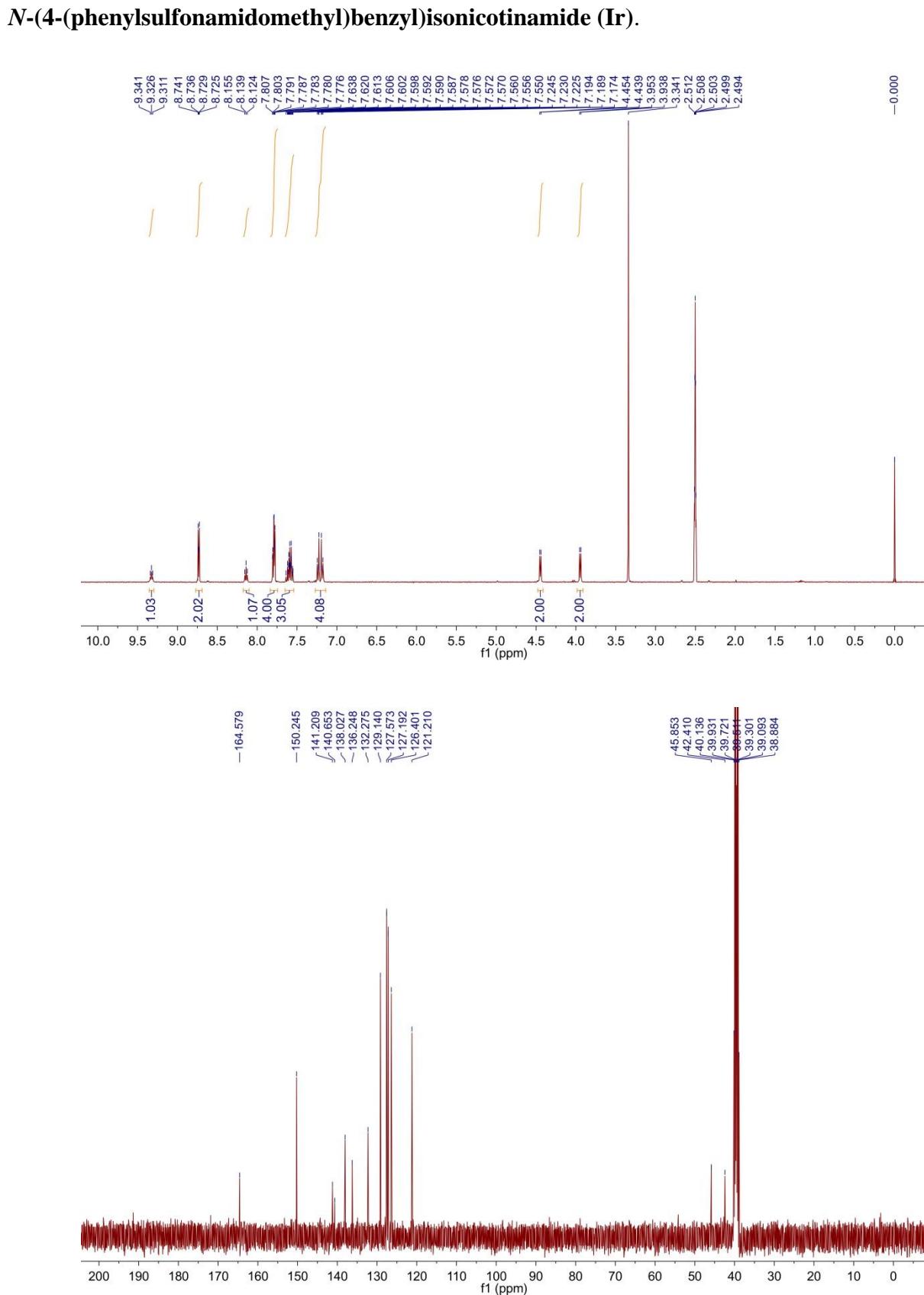
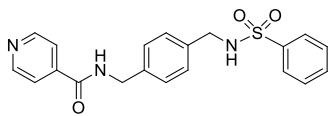
2,4,6-Trichloro-N-(4-(phenylsulfonamidomethyl)benzyl)benzamide (Ip).

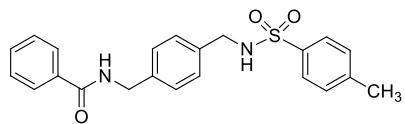




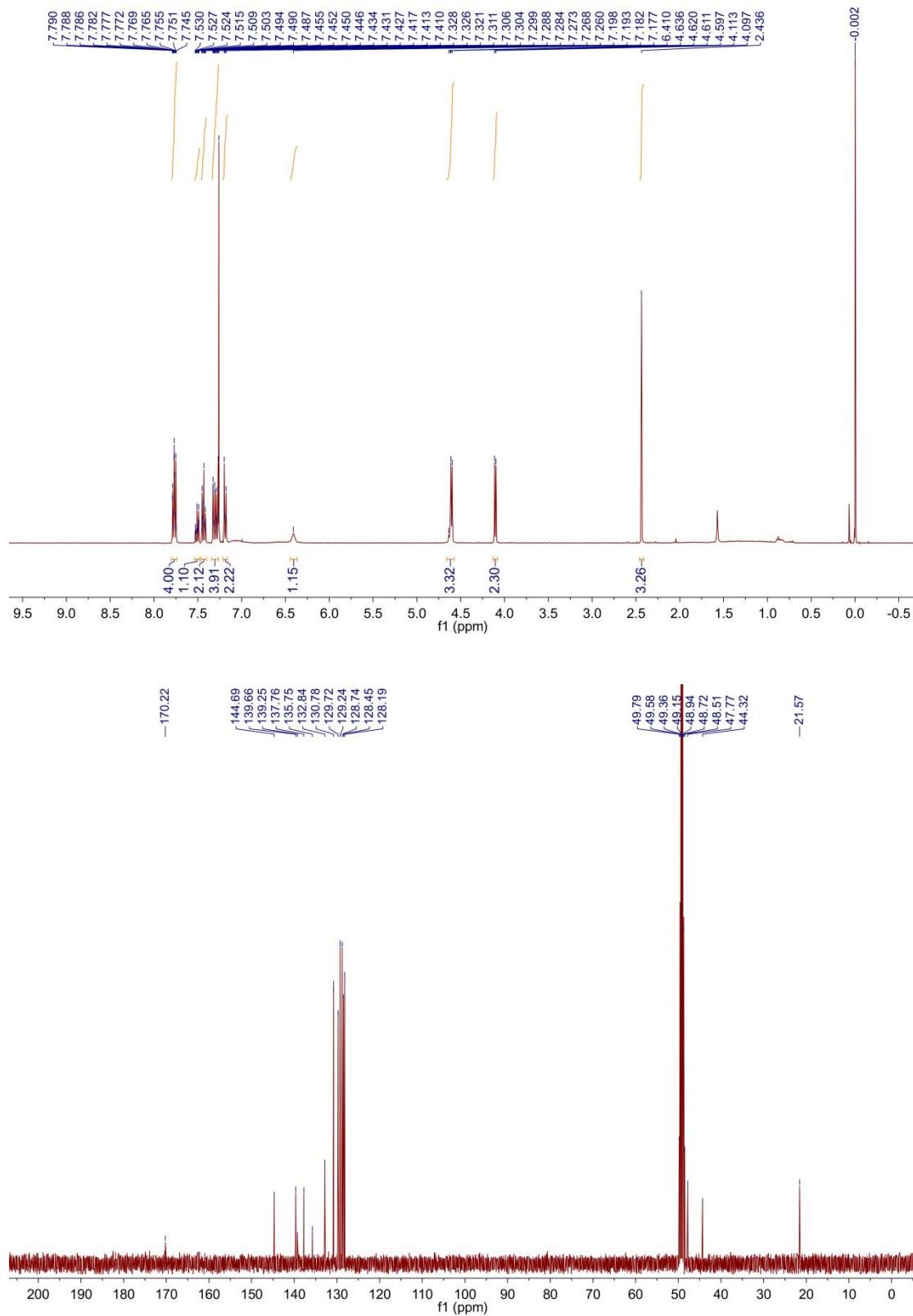
N-(4-(phenylsulfonamidomethyl)benzyl)nicotinamide (Iq).

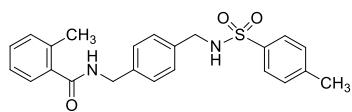




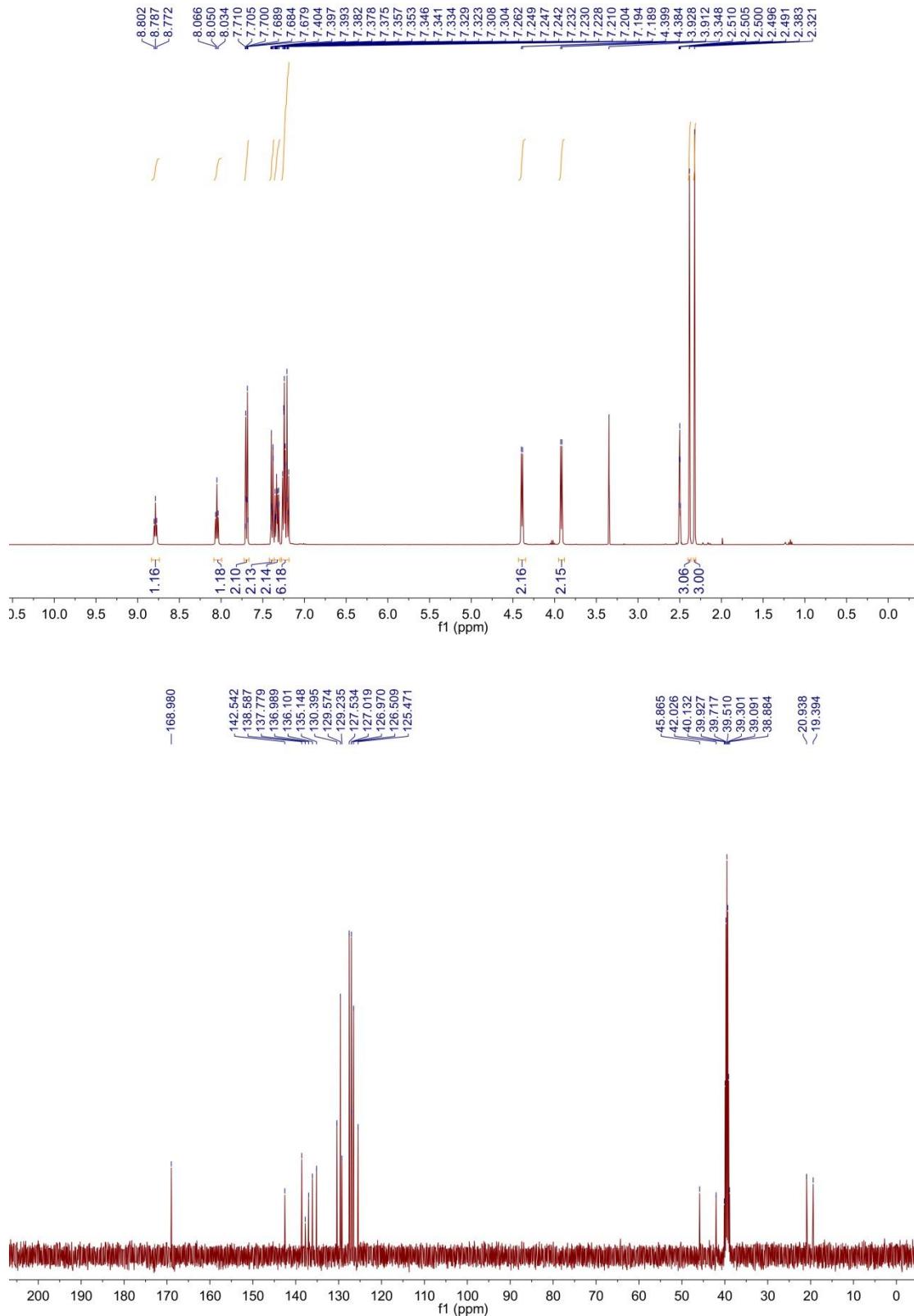


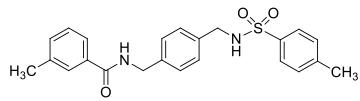
N-((4-methylphenylsulfonamido)methyl)benzyl)benzamide (IIa).



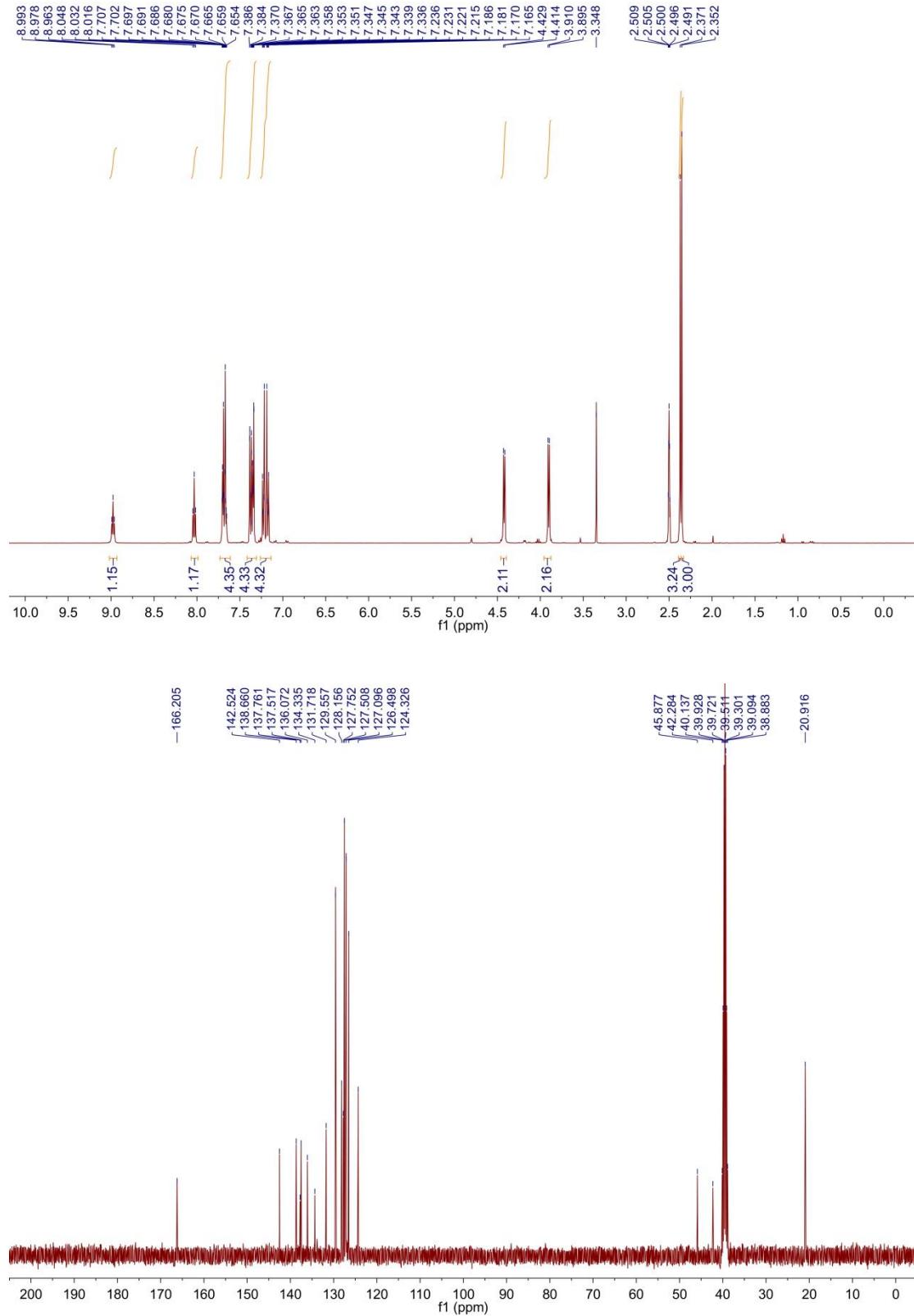


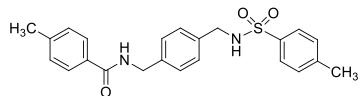
2-Methyl-N-(4-((4-methylphenylsulfonamido)methyl)benzyl)benzamide (IIb).



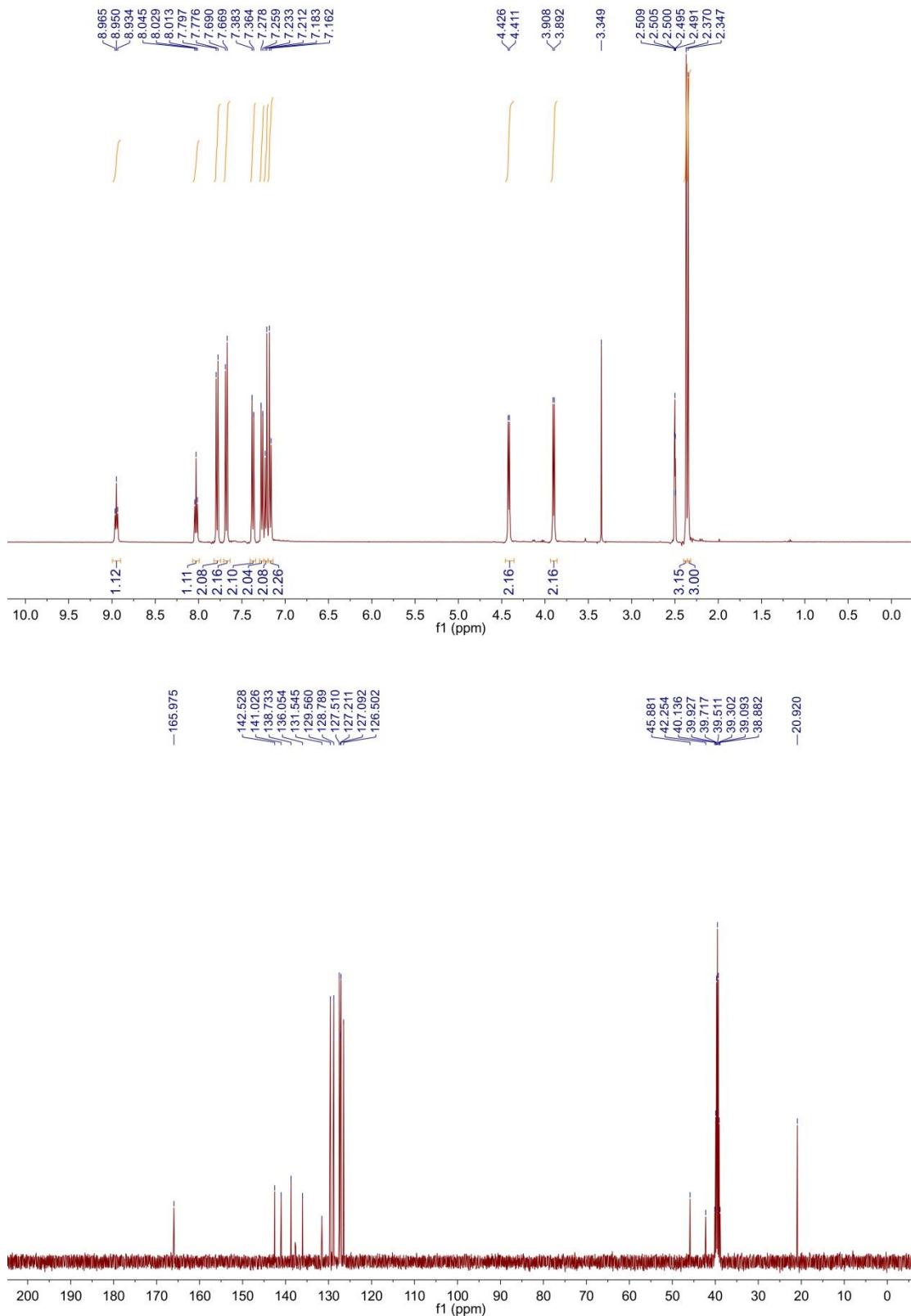


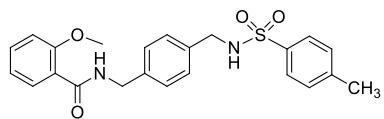
3-Methyl-N-(4-((4-methylphenylsulfonamido)methyl)benzyl)benzamide (IIc).



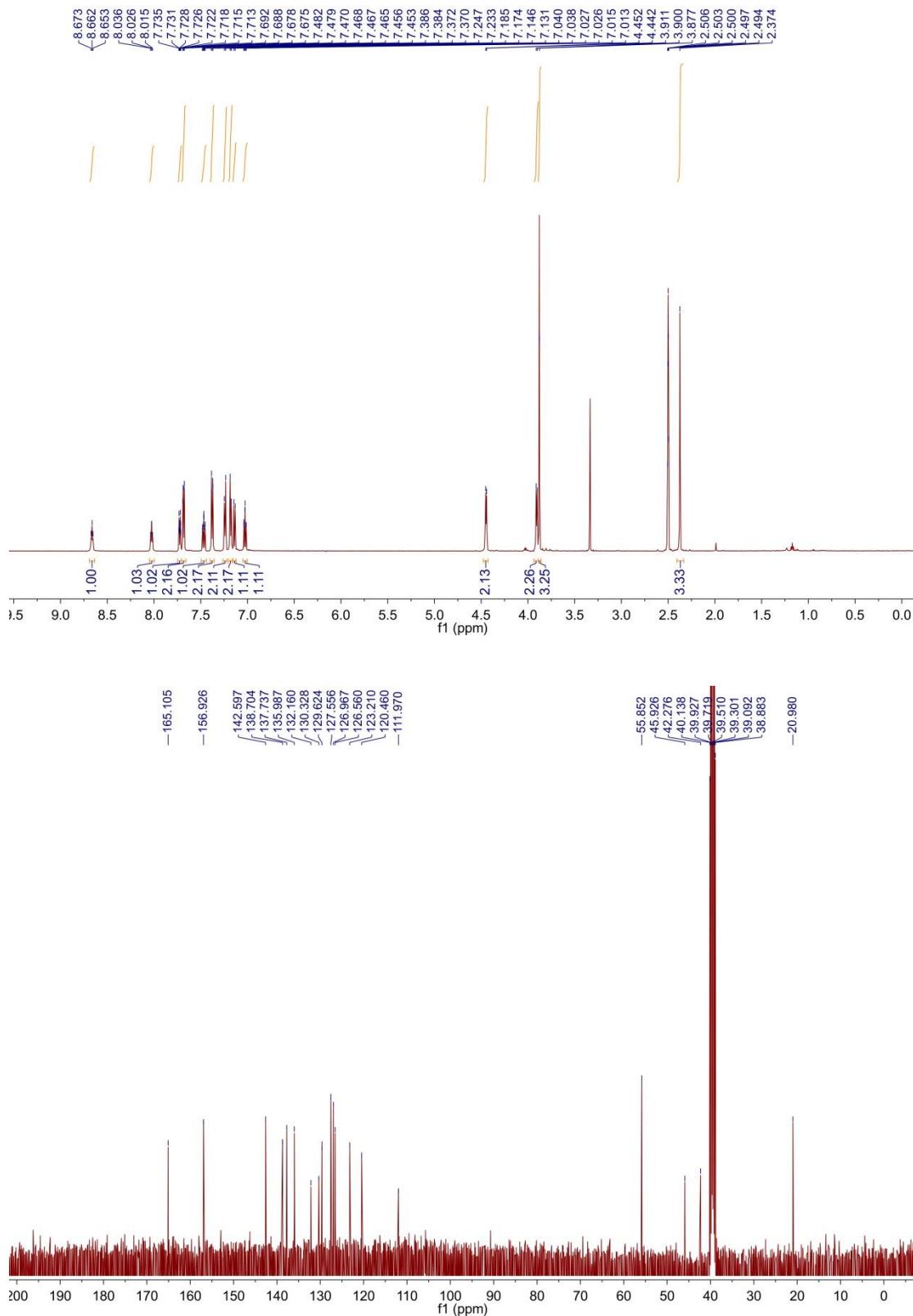


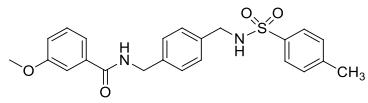
4-Methyl-N-(4-((4-methylphenylsulfonamido)methyl)benzyl)benzamide (IId).



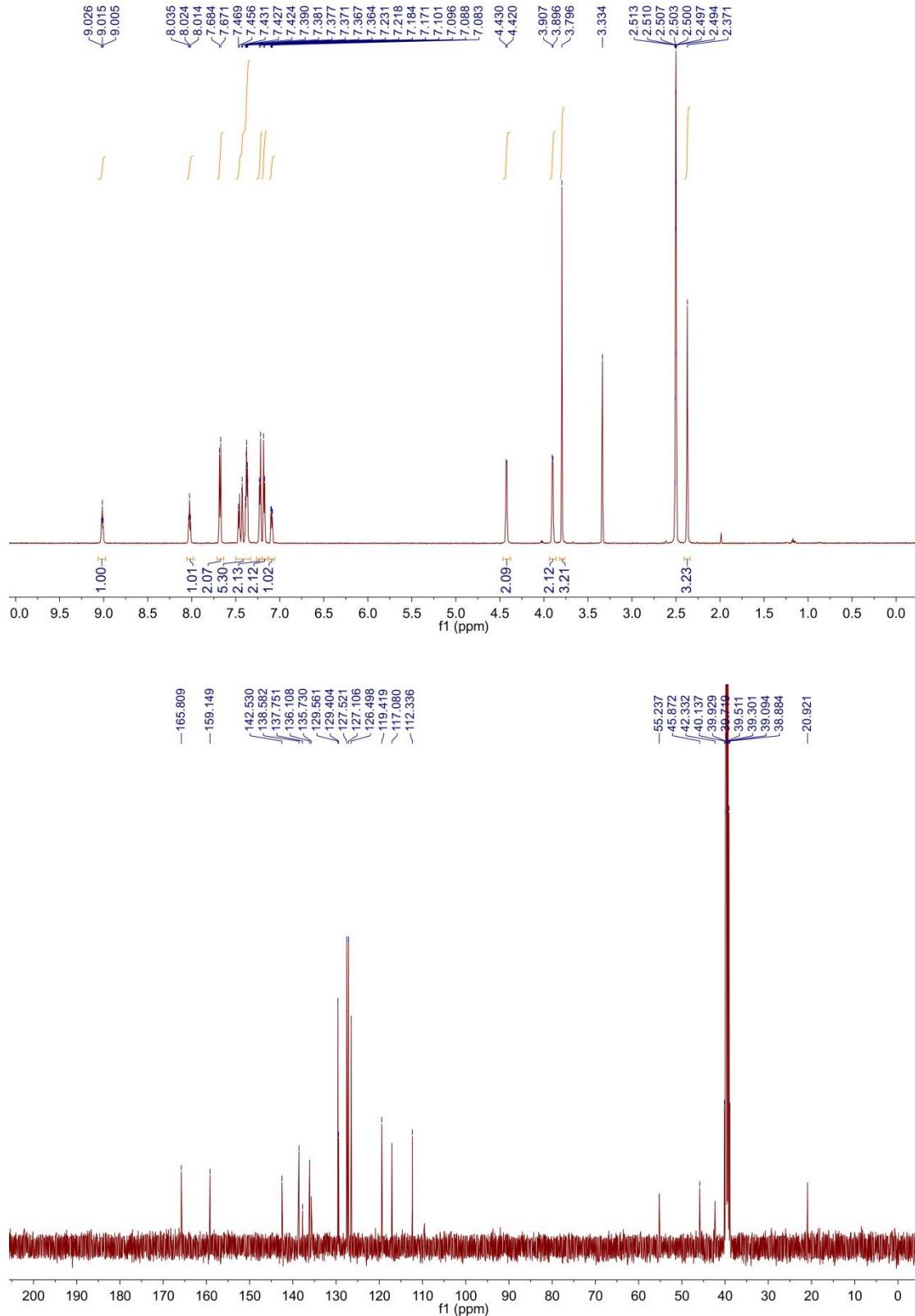


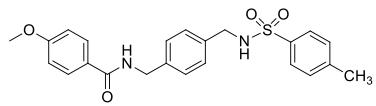
2-Methoxy-N-(4-((4-methylphenylsulfonamido)methyl)benzyl)benzamide (IIe).



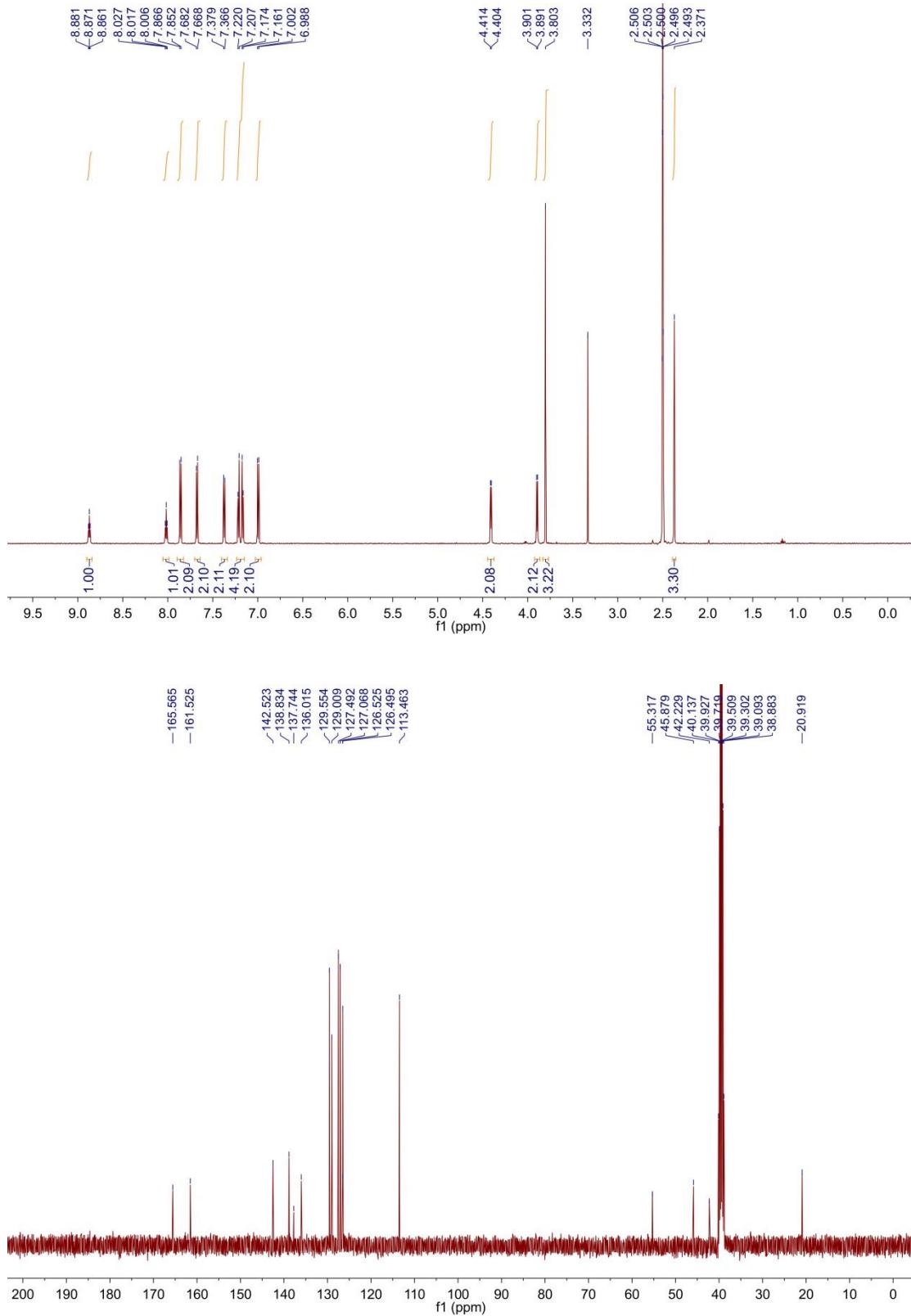


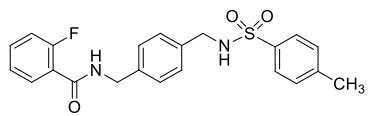
3-Methoxy-N-(4-((4-methylphenylsulfonamido)methyl)benzyl)benzamide (IIf).



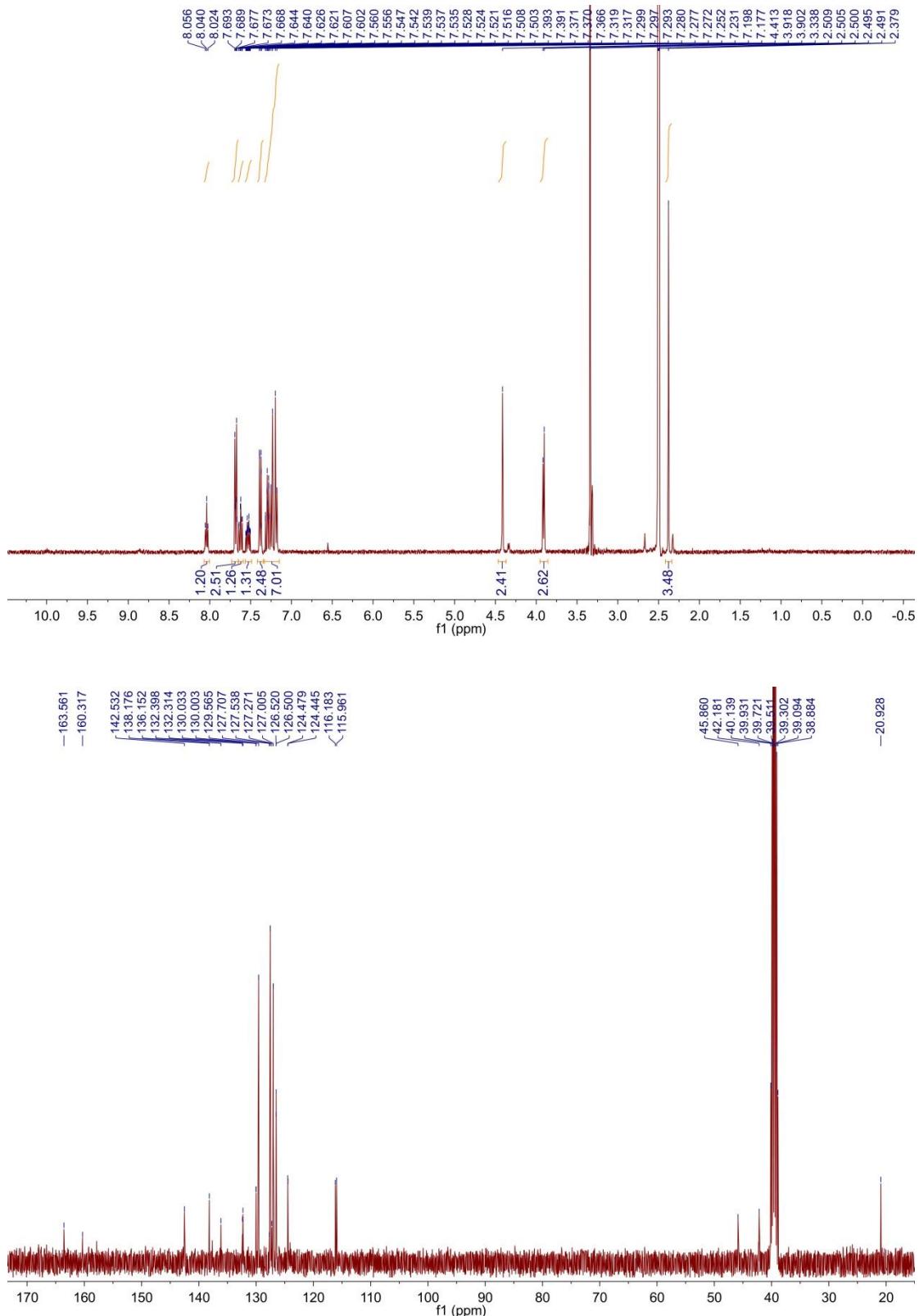


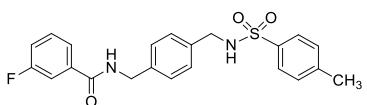
4-Methoxy-N-(4-((4-methylphenylsulfonamido)methyl)benzyl)benzamide (IIg).



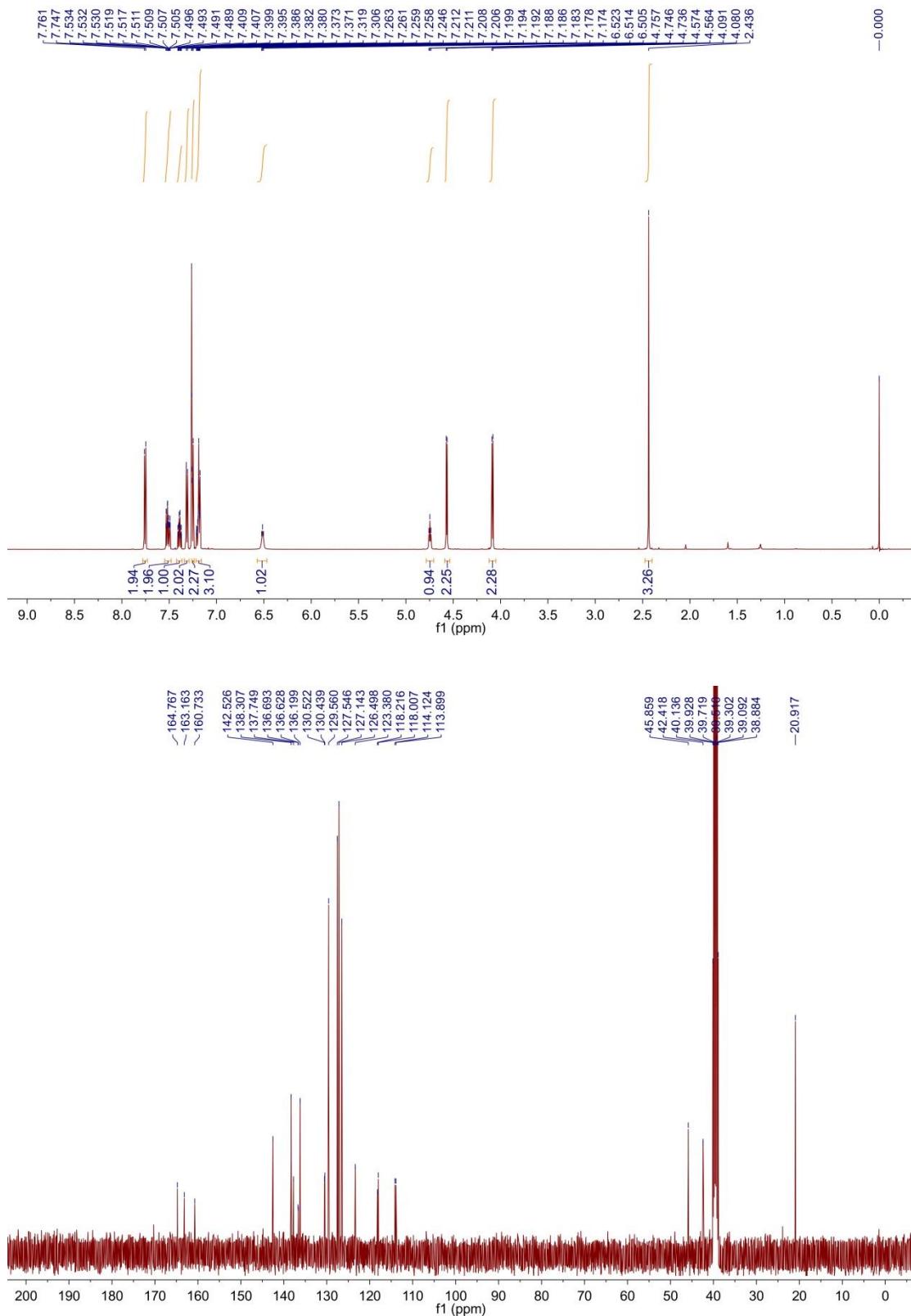


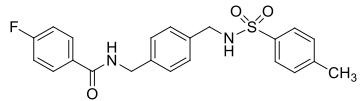
2-Fluoro-N-(4-((4-methylphenylsulfonamido)methyl)benzyl)benzamide (IIh).



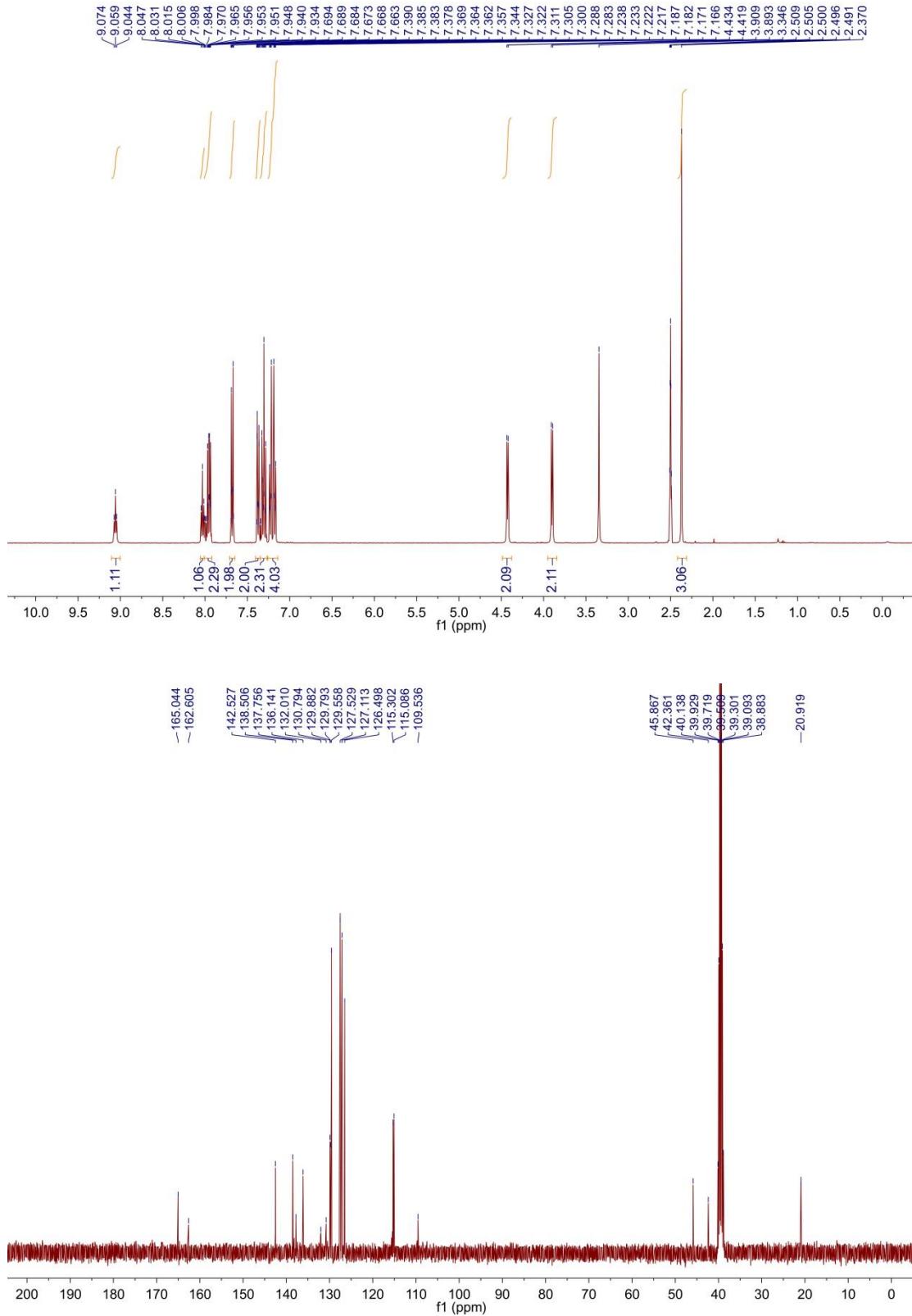


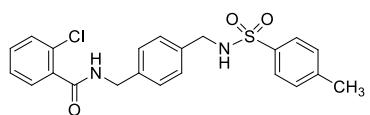
3-Fluoro-N-(4-((4-methylphenylsulfonamido)methyl)benzyl)benzamide (III).



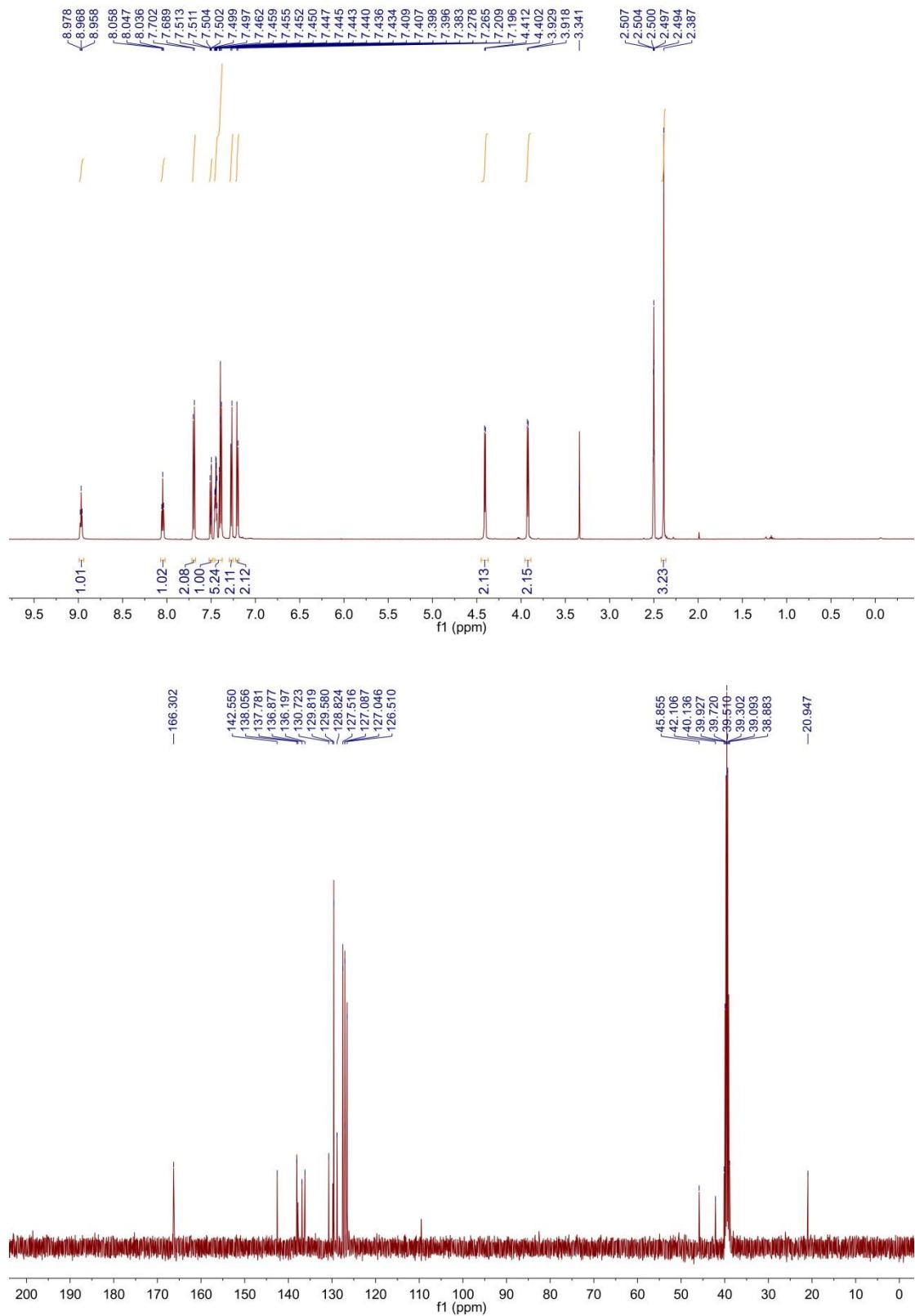


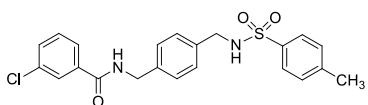
4-Fluoro-N-(4-((4-methylphenylsulfonamido)methyl)benzyl)benzamide (IIj).



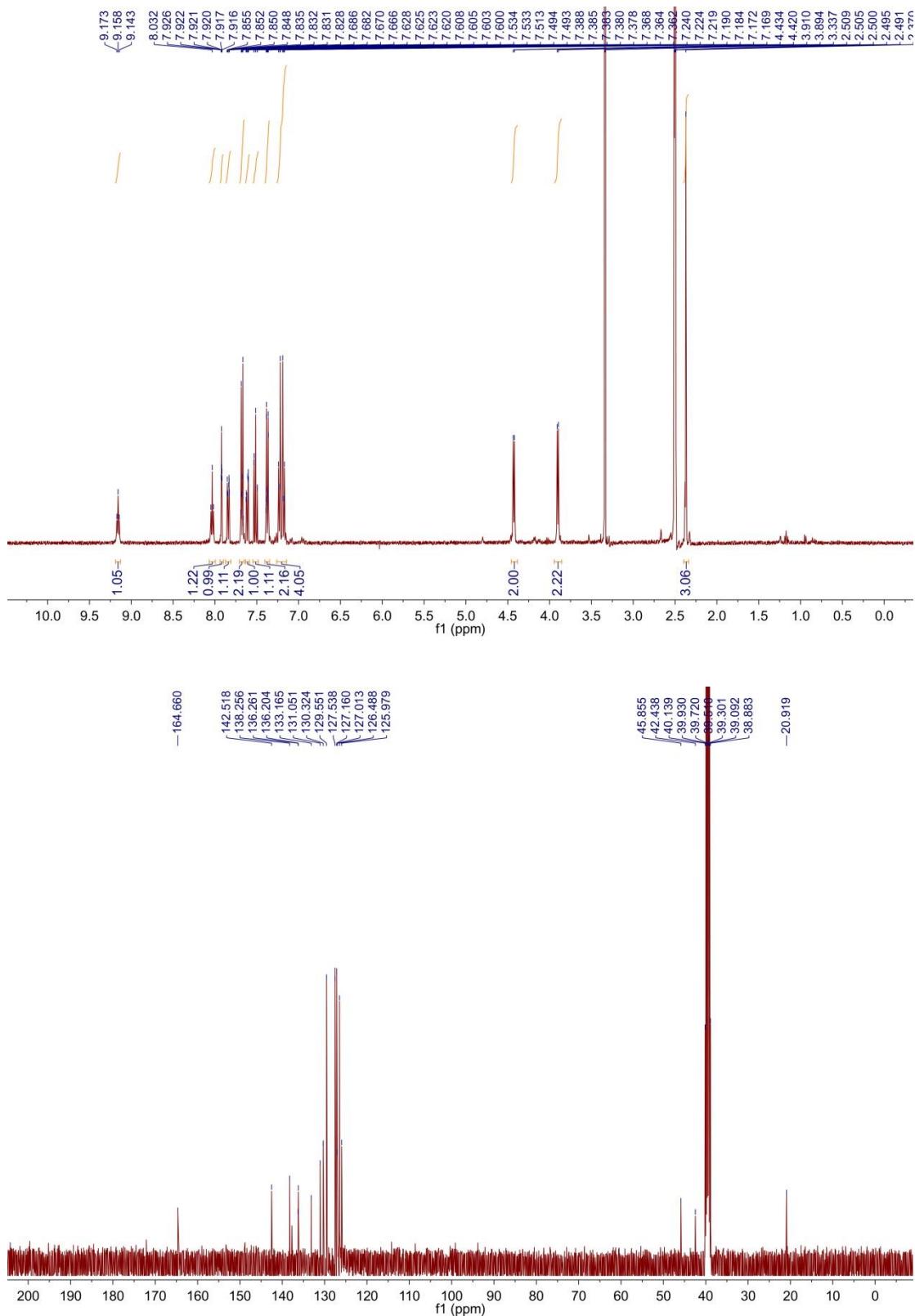


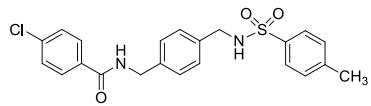
2-Chloro-N-(4-((4-methylphenylsulfonamido)methyl)benzyl)benzamide (IIk).



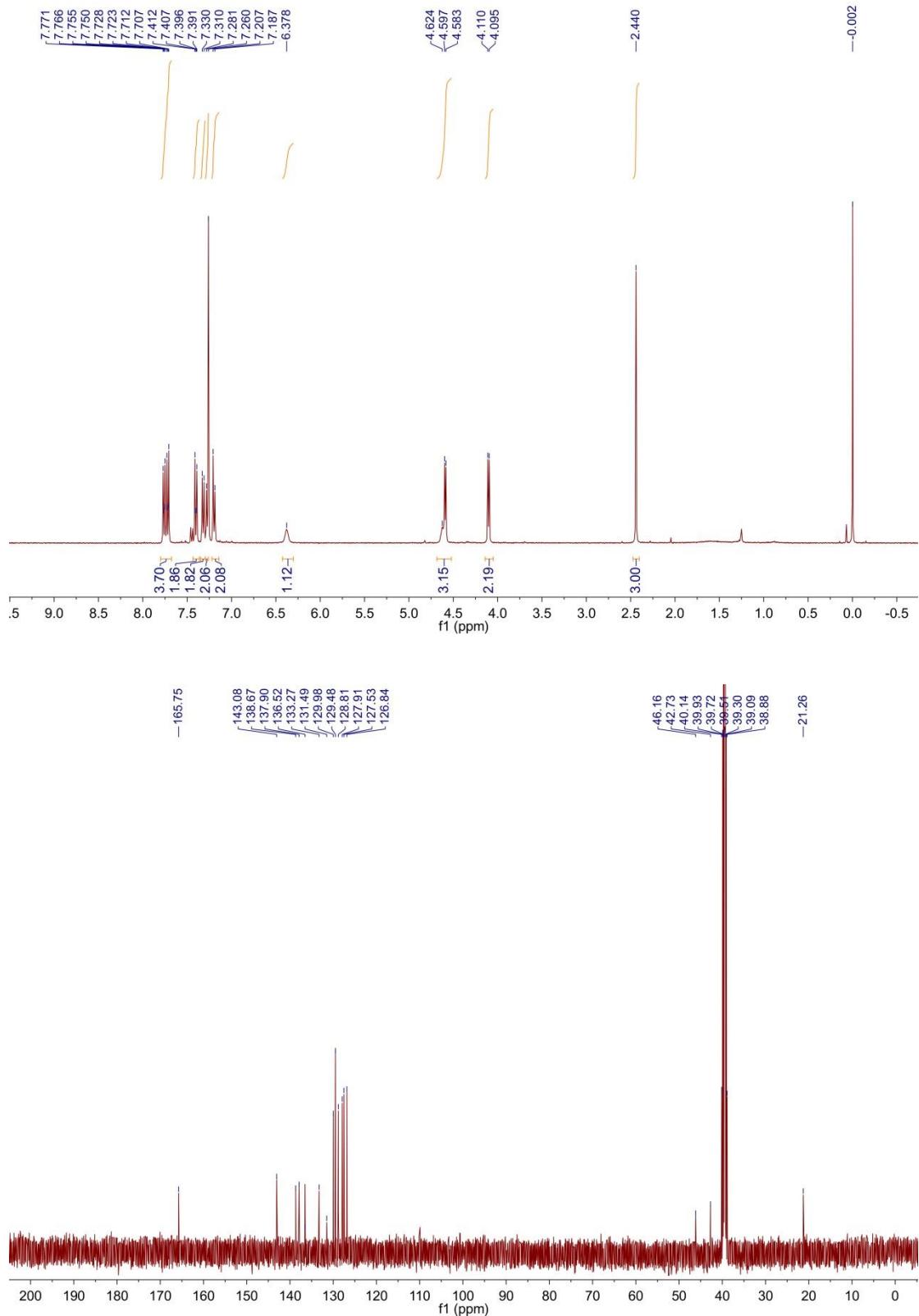


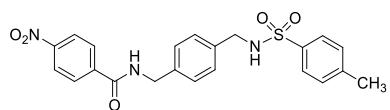
3-Chloro-N-(4-((4-methylphenylsulfonamido)methyl)benzyl)benzamide (IIm).



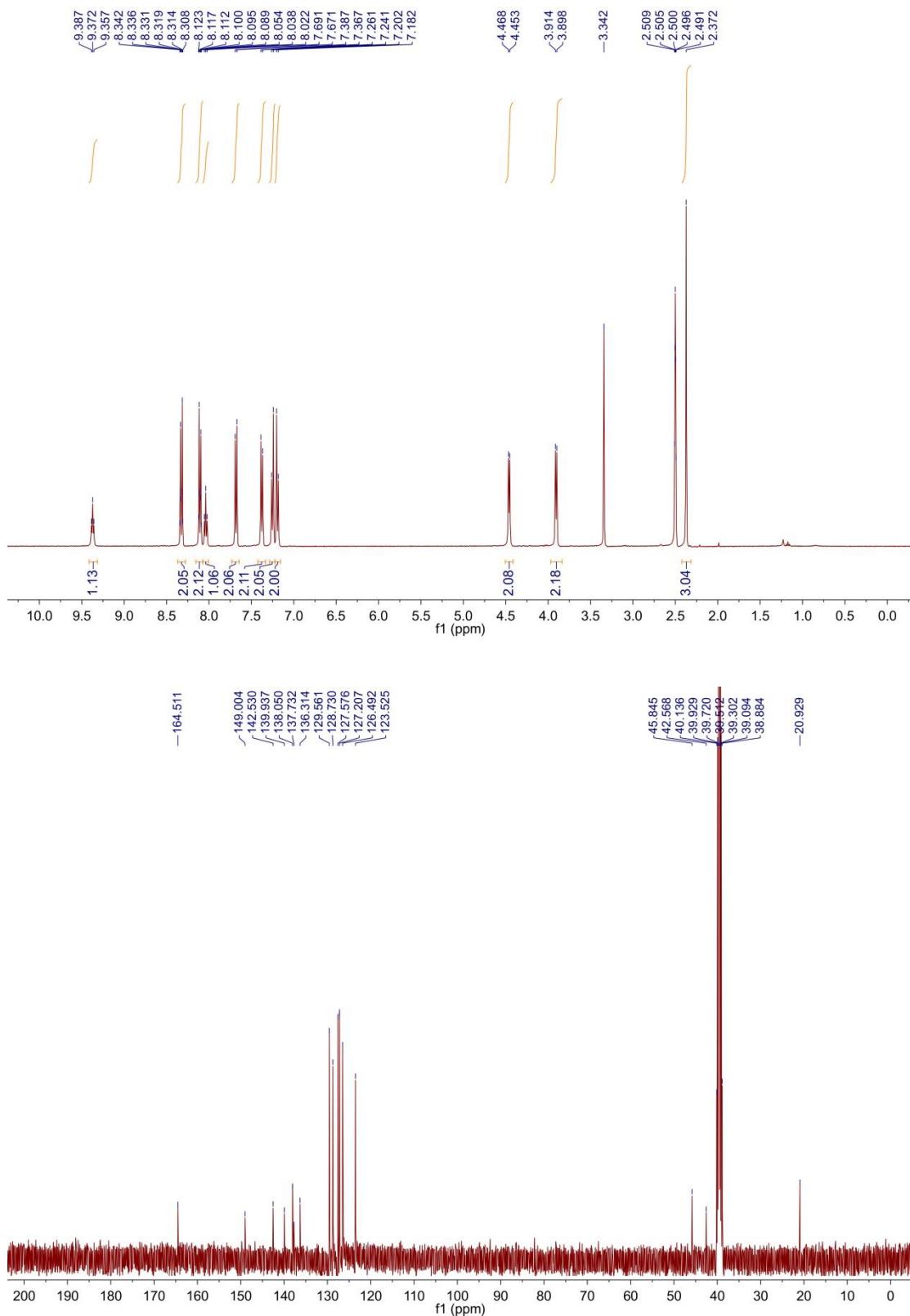


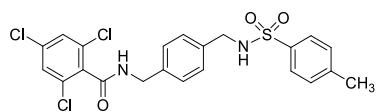
4-Chloro-N-(4-((4-methylphenylsulfonamido)methyl)benzyl)benzamide (IIIn).



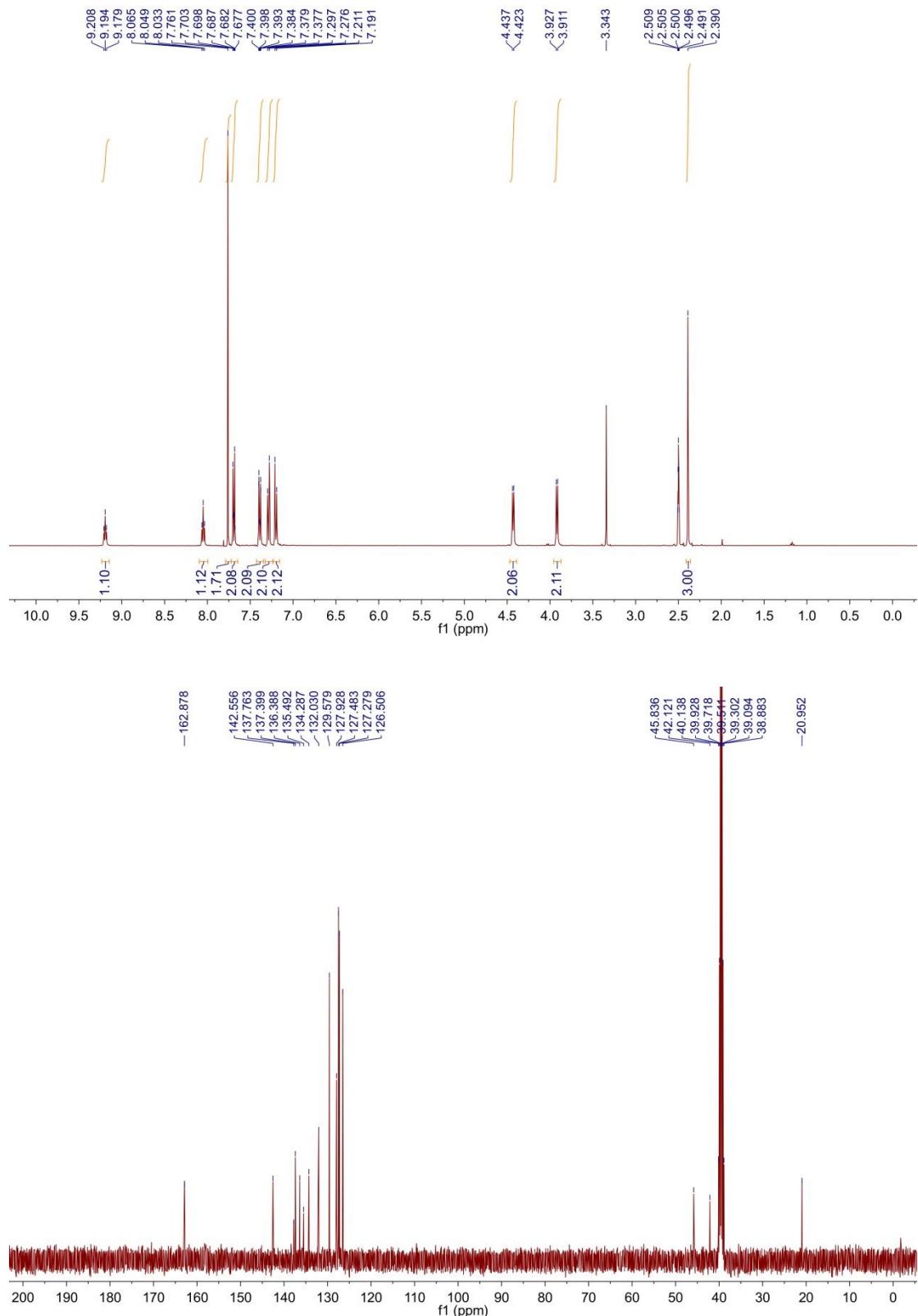


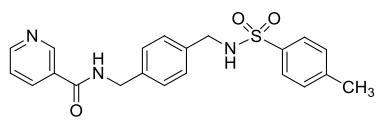
N-((4-methylphenylsulfonamido)methyl)benzyl)-4-nitrobenzamide (IIo).





2,4,6-Trichloro-N-(4-((4-methylphenylsulfonamido)methyl)benzyl)benzamide (IIp).





N-((4-methylphenylsulfonamido)methyl)benzylnicotinamide (IIq).

